

A Progress Report

July 1, 1990 to December 31, 1990

NASA-UVA LIGHT AEROSPACE ALLOY AND
STRUCTURES TECHNOLOGY PROGRAM

NASA-LaRC Grant NAG-1-745

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**NASA-UVA LIGHT AEROSPACE ALLOY
AND STRUCTURES TECHNOLOGY PROGRAM**

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NASA-UVa LIGHT AEROSPACE ALLOY AND STRUCTURES TECHNOLOGY PROGRAM

EXECUTIVE SUMMARY

The NASA-UVa Light Aerospace Alloy and Structures Technology Program (LA²ST) has achieved a high level of activity in 1990, with eleven projects being conducted by graduate students and faculty advisors in the Departments of Materials Science, Civil Engineering, and Mechanical and Aerospace Engineering at the University of Virginia. This work is funded by the NASA-Langley Research Center under Grant NAG-1-745. Here, we report on the progress achieved between July 1 and December 30, 1990.

The general objective of the LA²ST Program is to conduct interdisciplinary graduate student research on the performance of next generation, light weight aerospace alloys, composites and associated thermal gradient structures in close collaboration with Langley researchers. Specific technical objectives are established for each research project. We aim to produce basic understanding of material behavior and microstructure, new monolithic and composite alloys, advanced processing methods, new solid and fluid mechanics analyses, measurement advances and a pool of educated graduate students.

The accomplishments presented in this report are highlighted as follows:

- oo Four research areas are being actively investigated, including: (1) Mechanical and Environmental Degradation Mechanisms in Advanced Light Metals and Composites, (2) Aerospace Materials Science, (3) Mechanics of Materials and Composites for Aerospace Structures, and (4) Thermal Gradient Structures.
- oo Eleven research projects are being conducted by 6 PhD and 6 MS graduate students, 11 faculty members, and 2 research associates from four departments in the Engineering School at UVa. Each project is in conjunction with a specific branch and technical monitor at LaRC.
- oo Four undergraduate students, recruited from North Carolina State and California Polytechnic State Universities, worked at the Langley Research Center during the summer of 1990. Two undergraduates are assisting in LA²ST research projects at UVa.

- oo Reporting accomplishments between July and December of 1990 include 11 journal or proceedings publications and 11 presentations at technical meetings, bringing the LA²ST totals since 1986 to 26 and 34, respectively.
- oo One PhD student and three Masters level students graduated during this reporting period, and four theses/dissertations were published.
- oo ***Research on environmental fatigue of advanced aluminum alloys and metal matrix composites*** developed the experimental capabilities to characterize da/dN-ΔK for the rigidly gripped single edge cracked specimen and to monitor crack mouth opening displacement indicative of the extent of crack closure in aggressive environments. The first application of this method indicates that sheet forms of peak aged 2090 and 2091 have poor intrinsic FCP resistance compared to plate for moist air.
(Program 1)
- oo ***Research on localized corrosion and stress corrosion cracking of Al-Li-Cu alloy 2090 in aqueous solutions*** demonstrated the dominant roles of microstructural heterogeneity, localized environment chemistry and surface films. Short-transverse SCC proceeds along subgrain boundaries by anodic dissolution of T₁ precipitates. Alloy 2090 is susceptible to preexposure embrittlement. Pits retain electrolyte when removed from the bulk solution and become alkaline. When combined with CO₂ from moist air, this alkaline solution initiates reactions which accelerate SCC. The student on this program successfully graduated with the PhD degree in Materials Science at UVA.
(Program 5)
- oo ***Research on the environmental sensitivity of Al-Li-X alloys*** demonstrated that Zn additions to alloy 8090 result in an alteration of precipitate morphology and distribution. These changes likely influence SCC susceptibility. Breaking load measurements are being performed to evaluate and correlate these effects. This work is cosponsored by Alcoa Technical Center.
(Program 6)
- oo ***Research at VPI on hydrogen embrittlement of aluminum alloys*** employed the disk rupture method to show a decrease of the strain to failure for alloys 2090 and 2091 due to high pressure gaseous hydrogen compared to nitrogen. No important variations were seen for 2219 and Weldalite™ 049. Hydrogen had no effect on the cryogenic fracture behavior for each of the alloys. The student on this program attained the MS degree in Materials Engineering at VPI.
(Program 7)

- oo ***Research on the fracture behavior of Al-Cu-Li-In alloys*** demonstrated that, although indium promotes an increase in hardness and ultimate tensile strength for 2090 in the T6 condition, the yield strength of material from DC cast ingots is not increased for the unrecrystallized condition. The ambient temperature fracture toughness of 2090 + In-T6 is lower than commercially available 2090-T81 because of subgrain boundary precipitation and associated intersubgranular cracking that preempts slip band cracking and high angle boundary delamination. A J-integral fracture characterization capability was established at LaRC where this research project is being conducted. (Program 3)
- oo ***Research on the fracture toughness of Weldalite™ 049*** was initiated to examine the temperature dependencies of the crack initiation and growth toughnesses for two model microstructures containing either the T_1 phase or δ' plus T_1 . This research is being conducted at LaRC, in collaboration with a separate program at UVa (Program 4)
- oo ***Research on elevated temperature fracture of PM Al-Fe-Si-V alloy 8009*** demonstrated an intrinsic embrittlement phenomenon that is maximized at an intermediate temperature of 150 to 200°C. Two extrusions and a rolled plate of 8009 reproducibly exhibit this behavior. Embrittlement, in terms of initiation and growth toughnesses, increases with decreasing loading rate at 175°C. This phenomenon is likely for other fine grain size, rapidly solidified aluminum alloys. For 8009, delamination occurs for specimens from the extrusions, but is not prevalent in rolled plate, where toughness is isotropic due to a homogeneous prior ribbon boundary microstructure. (Program 2)
- oo ***Research on elevated temperature subcritical cracking in advanced Al alloys*** confirmed "creep crack growth" in PM alloy 8009 in moist air between 175 and 325°C. Cracking occurs at stress intensity levels well below K_{IC} and is pronounced at 175°C. 8009 is a creep brittle material; growth rates correlate with K and more precisely, with J . The creep crack growth parameters, C_I and C^* , do not describe cracking because crack tip creep is minimal; 8009 is not a creep ductile material. (Program 2)
- oo ***Research on Ti alloy matrix-SiC fiber reinforced composites*** determined the reactivities of six titanium matrices, including Ti-1100 and BETA 21S, with a commercial SiC fiber (SCS-6) at temperatures between 800 and 1000°C. Alloying slowed the rate of reaction in all cases studied. Life predictions were made from the kinetic data for temperatures of 700 to 1000°C. A mechanism for the growth of the reaction zone was proposed. The student on this program successfully graduated with the MS degree in Materials Science at UVa. (Program 8)

- oo ***Research on quantifying the spatial distribution and homogeneity of microstructure*** developed mathematical descriptions of particles in materials based on the Dirichlet tessellation technique. The analysis operates on a personal computer. A powder metallurgy alloy (Al-2Mn-1.2Si-0.4V-0.1Zr) was chosen for systematic study of the effects of processing parameters on the distribution of oxide particles in stringers. Processing, under variable conditions, will be conducted at NASA-LaRC and at UVA, mechanical properties will be determined at NASA and microstructural descriptions will be developed at UVA. (Program 9)
- oo ***Research on the yielding of SCS-6/Ti-15-3 MMC under biaxial loading*** obtained MMC tubes, designed and tested loading fixtures, enhanced and evaluated a data acquisition system, conducted preliminary experiments, and enhanced the capability for micromechanical modelling. (Program 10)
- oo ***Research on cryogenic tankage*** demonstrated that a proposed new structure for the tanks of large launch vehicles, in which stringers and frames are formed superplastically, is feasible. This problem was analyzed by finite element methods, with computer facilities at UVA and NASA-LaRC. During 1991, the research emphasis will be on thermal problems in tanks and shell structures for aerospace vehicles and high speed transports. The student on this program successfully graduated with the MS degree in Mechanical and Aerospace Engineering at UVA. (Program 11)
- oo ***Research on the thermoviscoplastic behavior*** of high temperature alloy panels demonstrated material and geometric nonlinearities. Preliminary tests with a stainless steel panel established the basic requirements for the experimental procedure. A refined test fixture was designed and fabricated. A test program was initiated with Hastelloy-X panels which were instrumented with thermocouples, strain gages and displacement gages. (Program 12)

INTRODUCTION

Background

Since 1986, the Metallic Materials Branch in the Materials Division of the NASA-Langley Research Center has sponsored graduate student engineering and scientific research at the University of Virginia and at Virginia Polytechnic Institute and State University in the Departments of Materials Science and Materials Engineering, respectively. This work has emphasized the mechanical and corrosion behavior of light aerospace alloys, particularly Al-Li based compositions, in aggressive aerospace environments^[1]. Initial results are documented in three progress reports^[2-4].

In the Fall of 1988 this program was increased in scope to incorporate materials science research at UVa on the development and processing of advanced aerospace materials^[5]. In early 1989 the program was further enhanced to include interdisciplinary work on solid mechanics and thermal structures, as funded by several Divisions within the Structures Directorate at NASA-LaRC^[6]. The Department of Civil Engineering and the Department of Mechanical and Aerospace Engineering at UVa participated in this expanded program. With this growth, the NASA-UVa Light Aerospace Alloy and Structures Technology Program (LA²ST) was initiated within the School of Engineering and Applied Science at UVa.

The first progress report for the LA²ST program was published in August of 1989^[7]. Research efforts in solid mechanics were in a state of infancy and were not represented at that time. Since then, graduate students have been recruited into the structural mechanics programs and several new projects have been initiated. Since July of 1989, the LA²ST program has operated with full participation from all faculty and graduate students, as outlined in the last two progress reports^[8,9] and two grant renewal proposals^[10,11]. The first two-day Grant Review Meeting was held in June of 1990 at the Langley Research Center, with over 20 faculty and graduate students from UVa and 1 faculty and graduate student from VPI participating.

This report summarizes the progress of LA²ST research during the period from July 1st to December 30, 1990.

Problem and Needs

Future aerospace structures require advanced light alloys and composites with associated processing and fabrication methods; new structural design methods and concepts with experimental evaluations; component reliability/durability/damage tolerance prediction procedures; and a pool of doctoral level engineers and scientists. Work on advanced materials and structures must be interdisciplinary and fully integrated. Nationally, academic efforts in these areas are limited. The NASA-UVa Light Aerospace Alloy and Structures Technology Program addresses these needs.

LA²ST Program

As detailed in the original proposal^[6] and affirmed in the most recent renewal document^[11], faculty from the Departments of Materials Science, Mechanical and Aerospace Engineering, and Civil Engineering at UVa are participating in the LA²ST research and education program focused on high performance, light weight, aerospace alloys and structures. We aim to develop long term and interdisciplinary collaborations between graduate students, UVa faculty, and NASA-Langley researchers.

Our research efforts are producing basic understanding of materials performance, new monolithic and composite alloys, advanced processing methods, solid and fluid mechanics analyses, and measurement advances. A major product of the LA²ST program is graduate students with interdisciplinary education and research experience in materials science, mechanics and mathematics. These advances should enable various NASA technologies.

The scope of the LA²ST Program is broad. Four research areas are being investigated, including:

- oo Mechanical and Environmental Degradation Mechanisms in Advanced Light Metals and Composites,
- oo Aerospace Materials Science,
- oo Mechanics of Materials and Composites for Aerospace Structures,
- oo Thermal Gradient Structures.

Eleven specific research projects are ongoing within these four areas and are reported here. These projects involve eleven faculty, two research associates and twelve graduate students. The majority of the graduate students are at the doctoral level (60%) and are citizens of the United States (over 90%). In each case the research provides the basis for the thesis or dissertation requirement of graduate studies at the University of Virginia. Research is conducted at either UVa or LaRC, and under the guidance of UVa faculty and NASA staff. Each project is developed in conjunction with a specific LaRC researcher. Participating students and faculty are closely identified with a NASA-LaRC branch.

A primary goal of the LA²ST Program is to foster interdisciplinary research. To this end, many of the research projects share a common focus on light and reusable aerospace structures which will be subjected to aggressive terrestrial and space environments; with emphasis on both cryogenic and elevated temperature conditions with severe thermal gradients typical of tankage structures.

Organization of Progress Report

This progress report first provides LA²ST Program administrative information including statistics on the productivity of faculty and student participants, a history of current and graduated students, and a list of ongoing projects with NASA and UVa advisors. Eleven sections summarize the specific technical accomplishments of each research project for the period from July 1st to December 30th of 1990. These reports describe project objective, approach, results, and immediate future plans. Appendices document grant sponsored publications and conference participation, and provide abstracts of technical papers which were published during this reporting period.

References

1. R.P. Gangloff, G.E. Stoner and M.R. Louthan, Jr., "Environment Assisted Degradation Mechanisms in Al-Li Alloys", University of Virginia, Proposal No. MS-NASA/LaRC-3545-87, October, 1986.

2. R.P. Gangloff, G.E. Stoner and R.E. Swanson, "Environment Assisted Degradation Mechanisms in Al-Li Alloys", University of Virginia, Report No. UVA/528266/MS88/101, January, 1988.
3. R.P. Gangloff, G.E. Stoner and R.E. Swanson, "Environment Assisted Degradation Mechanisms in Advanced Light Metals", University of Virginia, Report No. UVA/528266/MS88/102, June, 1988.
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5. T.H. Courtney, R.P. Gangloff, G.E. Stoner and H.G.F. Wilsdorf, "The NASA-UVa Light Alloy Technology Program", University of Virginia, Proposal No. MS NASA/LaRC-3937-88, March, 1988.
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8. R.P. Gangloff, "NASA-UVa Light Aerospace Alloy and Structures Technology Program", University of Virginia, Report No. UVA/528266/MS90/105, December, 1989.
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10. R.P. Gangloff, "NASA-UVa Light Aerospace Alloy and Structures Technology Program", University of Virginia, Proposal No. MS NASA/LaRC-4512-90, November, 1989.
11. R.P. Gangloff, "NASA-UVa Light Aerospace Alloy and Structures Technology Program", University of Virginia, Proposal No. MS NASA/LaRC-4841-91, September, 1990.

SUMMARY STATISTICS

Table I documents the numbers of students and faculty who participated in the LA²ST Program, both during this reporting period and since the program inception in 1986. Academic and research accomplishments are indicated by the degrees awarded, publications and presentations. Specific graduate students and research associates who participated in the LA²ST Program are named in Tables II and III, respectively.

TABLE I: LA²ST Program Statistics

	<u>Current 7/1 to 12/30/90</u>	<u>Cumulative 1986 to 12/30/90</u>
PhD Students--UVa:	5	7
--NASA-LaRC:	1	1
MS Students--UVa:	4	4
--NASA:	1	1
--VPI:	1	1
Undergraduates--UVa	2	2
--NASA-LaRC	4	4
Faculty--UVa:	10	10
--VPI:	1	1
Research Associates--UVa:	2	2
PhD Awarded:	1	3

TABLE I: LA²ST Program Statistics (continued)

	<u>Current</u> <u>7/1 to 12/30/90</u>	<u>Cumulative</u> <u>1986 to 12/30/90</u>
MS Awarded:	3	3
Employers--NASA:	0	1
--Federal:	1	2
--University:	0	0
--Industry:	0	0
Publications:	11	26
Presentations:	11	34
Dissertations/Theses:	4	6
NASA Reports:	2	9

TABLE II
GRADUATE STUDENT PARTICIPATION IN THE NASA-UVA L²SI PROGRAM
January, 1991

POS #	GRADUATE STUDENT EMPLOYER	ENTERED PROGRAM	DEGREE COMPLETED	LANGLEY RESIDENCY	RESEARCH TOPIC	UVA/NASA-LaRC ADVISORS
1.	R. S. Piascik NASA-Langley	6/86	Ph.D. 10/89		Damage Localization Mechanisms in Corrosion Fatigue of Aluminum-Lithium Alloys	R. P. Gangloff D. L. Dicus
2.	J. P. Moran NIST	9/88	Ph.D. 12/89		An Investigation of the Localized Corrosion and Stress Corrosion Cracking Behavior of Alloy 2090	G. E. Stoner W. B. Lisagor
3.	R. G. Buchheit Sandia National Laboratories	6/87	Ph.D. 12/90		Measurements and Mechanisms of Localized Aqueous Corrosion in Aluminum-Lithium Alloys	G. E. Stoner D. L. Dicus
4.	D. B. Gundel Ph.D.-UVA	9/88	M.S. 12/90		Investigation of the Reaction Kinetics Between SiC Fibers and Titanium Matrix Composites	F. E. Wagner W. Brewer
5.	F. Rivet (VPI)	9/88	M.S. 12/90		Deformation and Fracture of Aluminum-Lithium Alloys: The Effect of Dissolved Hydrogen	R. E. Swanson (VPI) D. L. Dicus
6.	C. Copper Ph.D.-UVA	4/89	M.S. 12/90		Design of Cryogenic Tanks for Space Vehicles	W. D. Pilkey J. K. Haviland D. R. Rummel M.J. Shuart
7.	J. A. Wagner NASA-Langley	6/87	Ph.D. (12/91)	PhD Research @ LaRC	Temperature Effects on the Deformation and Fracture of Al-Li-Cu-In Alloys	R. P. Gangloff W. B. Lisagor J. C. Newman
8.	W. C. Porr, Jr.	1/88	Ph.D. (12/91)		Elevated Temperature Fracture of an Advanced Powder Metallurgy Aluminum Alloy	R. P. Gangloff C. E. Harris

TABLE II (continued)
GRADUATE STUDENT PARTICIPATION IN THE NASA-UVA LA² ST PROGRAM
 (continued)

<u>POS #</u>	<u>GRADUATE STUDENT EMPLOYER</u>	<u>ENTERED PROGRAM</u>	<u>DEGREE COMPLETED</u>	<u>LANGLEY RESIDENCY</u>	<u>RESEARCH TOPIC</u>	<u>UVA/NASA-LaRC ADVISORS</u>
9.	J. B. Parse	9/88	Ph.D. (5/91)		Quantitative Characterization of the Spatial Distribution of Particles in Materials	J. A. Wert D. R. Tenney
10.	D. C. Slavik	9/89	Ph.D. (12/92)		Environment Enhanced Fatigue of Advanced Aluminum Alloys and Composites	R. P. Gangloff D. L. Dicus
11.	C. L. Lach NASA-Langley	9/89	M.S. (12/91)	MS Research @LaRC	Effect of Temperature on the Fracture Toughness of Weldalite TM 049	R.P. Gangloff W. B. Lisagor
12.	R. J. Kilmer	11/89	Ph.D. (12/92)		Effect of Zn Additions on the Environmental Stability of Alloy 8090	G. E. Stoner W. B. Lisagor
13.	M. F. Coyle	12/89	M.S. (12/92)		Visoplastic Response of High Temperature Structures	E. A. Thornton J.H. Starnes
14.	C.J. Lissenden	9/90	Ph.D. (9/93)		Inelastic Response of Metal Matrix Composites Under Biaxial Loading	C.T. Herakovich M.J. Pindera W.S. Johnson

TABLE III
Post-Doctoral Research Associate Participation
in NASA-UVA LAST Program

<u>Pos</u> <u>#</u>	<u>Res. Assoc.</u> <u>Employer</u>	<u>Tenure</u>	<u>Research</u>	<u>Supervisor</u>
1.	Yang Leng	3/89 to 3/91	Elevated Tempera- ture Deformation and Fracture of PM Al Alloys and Composites	R. P. Gangloff
2.	Farshad Mizadeh	7/89 to 12/90	Deformation of Metal Matrix Composites	C. T Herakovich and Marek-Jerzy Pindera

COMPLETED PROJECTS

1. **DAMAGE LOCALIZATION MECHANISMS IN CORROSION FATIGUE OF ALUMINUM-LITHIUM ALLOYS**
 Faculty Investigator: R.P. Gangloff
 Graduate Student: Robert S. Piascik
 Degree: PhD
 UVa Department: Materials Science
 NASA-LaRC Contact: D. L. Dicus (Metallic Materials)
 Start Date: June, 1986
 Completion Date: November, 1989
 Employment: NASA-Langley Research Center

2. **AN INVESTIGATION OF THE LOCALIZED CORROSION AND STRESS CORROSION CRACKING BEHAVIOR OF ALLOY 2090 (Al-Li-Cu)**
 Faculty Investigator: Glenn E. Stoner
 Graduate Student: James P. Moran
 Degree: PhD
 UVa Department: Materials Science
 NASA-LaRC Contact: W.B. Lisagor (Metallic Materials)
 Start Date: September, 1988
 Completion Date: December, 1989
 Co-Sponsor: ALCOA
 Employment: National Institute of Standards and
 Technology

3. **MECHANISMS OF LOCALIZED CORROSION IN AL-LI-CU ALLOY 2090**
 Faculty Investigator: G.E. Stoner
 Graduate Student: R.G. Buchheit
 Degree: PhD
 UVa Department: Materials Science
 NASA-LaRC Contact: D.L. Dicus (Metallic Materials)
 Start Date: June, 1987
 Completion Date: December, 1990
 Cosponsor: Alcoa
 Employment: Sandia National Laboratories

4. DEFORMATION AND FRACTURE OF ALUMINUM-LITHIUM ALLOYS: THE EFFECT OF DISSOLVED HYDROGEN
Faculty Investigator: R.E. Swanson (VPI)
Graduate Student: Frederic C. Rivet
Degree: MS
VPI Department: Materials Engineering at VPI
NASA-LaRC Contact: D.L. Dicus (Metallic Materials)
Start Date: September, 1988
Completion Date: December, 1990
Employment: Not determined

5. INVESTIGATION OF THE REACTION KINETICS BETWEEN SiC FIBERS AND SELECTIVELY ALLOYED TITANIUM MATRIX COMPOSITES AND DETERMINATION OF THEIR MECHANICAL PROPERTIES
Faculty Investigator: F.E. Wawner
Graduate Student: Douglas B. Gundel; MS candidate
UVa Department: Materials Science
NASA-LaRC Contact: D.L. Dicus and W.B. Brewer (Metallic Materials)
Start Date: January, 1989
Completion Date: December, 1990
Employment: Graduate School, University of Virginia; PhD candidate on LA²ST Program; Department of Materials Science

6. DESIGN OF CRYOGENIC TANKS FOR SPACE VEHICLES
Faculty Investigators: W.D. Pilkey and J.K. Haviland
Graduate Student: Charles Copper; PhD candidate
UVa Department: Mechanical and Aerospace Engineering
NASA-LaRC Contact: Drs. D.R. Rummeler (Structural Mechanics Division), R.C. Davis and M.J. Shuart (Aircraft Structures)
Start Date: April, 1989
Completion Date: December, 1990
Employment: Graduate School, University of Virginia; PhD candidate on LA²ST Program; Department of Mechanical and Aerospace Engineering

ADMINISTRATIVE PROGRESS

Faculty Participation

Professor John R. Scully, a newly hired Assistant Professor in the Materials Science Department at UVa, has joined the LA²ST Program. Professor Scully previously worked at Sandia National Laboratories and is a specialist in electrochemistry, hydrogen-metal interactions and environmental fracture. He has proposed an effort on hydrogen embrittlement of aluminum alloys which will begin during the next reporting period.

Professor Robert E. Swanson has left the Materials Engineering Department at VPI and will no longer participate in the LA²ST program. Dr. Swanson advised Mr. Frederic C. Rivet who recently obtained the MS degree in Materials Engineering at VPI.

Student Recruitment

The LA²ST Program continues to attract a sufficient number of high quality graduate students to achieve our education and research objectives (Table III). One new graduate student, Mr. Clifford Lissenden, was recruited into the LA²ST Program during the Fall of 1990. Notably, two students (Messrs Copper and Gundel) graduated with the Master of Science Degree at UVa, and elected to continue graduate studies toward the PhD degree within the LA²ST Program. Professor Scully is interviewing students to fill a single open slot on his hydrogen embrittlement program.

Six undergraduate students were incorporated into the LA²ST Program during this reporting period. This increase in program scope was suggested by W.B. Lisagor of the Metallic Materials Branch and was detailed in a proposal to NASA-LaRC in April of 1990^[1]. During the summer of 1990, four students, recruited from North Carolina State and California Polytechnic State University, worked in the Metallic Materials (3 students) and Mechanics of Materials Branches (1 student) at the Langley Research Center. These undergraduates typically have completed three years of course work in metallurgy and materials science departments and have cumulative grade point averages between 3.0 and 3.5 (A = 4.0). Without exception, each student effectively contributed to LaRC engineering and

research programs, and gained important appreciation for aerospace materials and mechanics research. Two of these students expressed an interest in graduate school at UVa, and are candidates for the LA²St Program should they decide to enter the Engineering School at UVa.

Two UVa undergraduates are currently working on ongoing LA²ST projects in the Mechanical and Aerospace Engineering Department. It is likely that at least one of these students will enroll in graduate studies at UVa. Undergraduate participation in the LA²ST Program will continue during 1991. A brochure is being prepared to nationally advertise this opportunity in order to attract the best students for both NASA-LaRC summer employment and future graduate studies.

Complementary Programs at UVa

The School of Engineering and Applied Science at UVa has targeted materials and structures research for aerospace applications as an important area for broad future growth. The LA²ST Program is an element of this thrust. Several additional programs are of benefit to LA²ST work.

The Board of Visitors at UVa awarded SEAS an Academic Enhancement Program Grant in the area of Light Thermal Structures. The aim is to use University funds to seed the establishment of a world-class center of excellence which incorporates several SEAS Departments. This program is lead by Professor Thornton and should directly benefit NASA.

The Light Metals Center has existed within the Department of Materials Science at UVa for the past several years under the leadership of Professor H.G.F. Wilsdorf. A Virginia Center for Innovative Technology Development Center in Electrochemical Science and Engineering was established in 1988 with Professor G.E. Stoner as Director. Professors Pilkey, Thornton and Gangloff are conducting research under NASA-Headquarters Grant sponsorship to examine "Advanced Concepts for Metallic Cryo-thermal Space Structures"^[2,3]. Research within this program is complementing LA²ST studies.

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1. R.P. Gangloff, "NASA-UVa Light Aerospace Alloy and Structures Technology Program: A Supplementary Proposal", University of Virginia, Proposal No. MS NASA/LaRC-4677-90, April, 1990.
2. W.P. Pilkey, "Advanced Concepts for Metallic Cryo-thermal Space Structures", University of Virginia Proposal No. MAE-NASA/HQ-4462-90, August, 1989.
3. W.P. Pilkey, "Advanced Concepts for Metallic Cryo-thermal Space Structures", University of Virginia Report No. UVA/528345/MAE91/101, February, 1991.

CURRENT PROJECTS

MECHANICAL AND ENVIRONMENTAL DEGRADATION MECHANISMS IN ADVANCED LIGHT METALS AND COMPOSITES

1. ENVIRONMENT-ENHANCED FATIGUE OF ADVANCED ALUMINUM ALLOYS AND METAL MATRIX COMPOSITES*
Faculty Investigator: R.P. Gangloff
Graduate Student: Donald Slavik; PhD Candidate
UVa Department: Materials Science
NASA-LaRC Contact: D.L. Dicus (Metallic Materials)
Start Date: September, 1989
Anticipated Completion Date: December, 1992
Program # 1

2. ELEVATED TEMPERATURE FRACTURE OF AN ADVANCED RAPIDLY SOLIDIFIED, POWDER METALLURGY ALUMINUM ALLOY*
Faculty Investigator: R.P. Gangloff
Graduate Student: William C. Porr, Jr.; PhD candidate
UVa Department: Materials Science
NASA-LaRC Contact: C.E. Harris (Mechanics of Materials)
Start Date: January, 1988
Anticipated Completion Date: December, 1991
Program # 2

3. ELEVATED TEMPERATURE CRACK GROWTH IN ADVANCED ALUMINUM ALLOYS*
Faculty Investigator: R. P. Gangloff
Research Associate: Dr. Yang Leng
Graduate Student: None
UVa Department: Materials Science
NASA-LaRC Contact: C.E. Harris (Mechanics of Materials)
Start Date: March, 1989
Anticipated Completion Date: July, 1991
Program # 2

4. TEMPERATURE EFFECTS ON THE DEFORMATION AND FRACTURE OF AL-Li-Cu-In ALLOYS*
Faculty Investigator: R.P. Gangloff
Graduate Student: John A. Wagner; PhD candidate and NASA-LaRC employee
UVa Department: Materials Science
NASA-LaRC Contacts: W.B. Lisagor (Metallic Materials)
J.C. Newman (Mechanics of Materials)
Start Date: June, 1987
Anticipated Completion Date: May, 1992
Program # 3
5. THE EFFECT OF TEMPERATURE ON THE FRACTURE TOUGHNESS OF WELDALITE™ *
Faculty Investigator: R.P. Gangloff
Graduate Student: Cynthia L. Lach; MS candidate and NASA-LaRC employee
UVa Department: Materials Science
NASA-LaRC Contacts: W.B. Lisagor (Metallic Materials)
Start Date: August, 1990
Anticipated Completion Date: May, 1992
Program # 4
6. MEASUREMENTS AND MECHANISMS OF LOCALIZED CORROSION IN AL-LI-CU ALLOYS*
Faculty Investigator: G.E. Stoner
Graduate Student: R.G. Buchheit
UVa Department: Materials Science
NASA-LaRC Contact: D.L. Dicus (Metallic Materials)
Start Date: June, 1987
Completion Date: December, 1990
Cosponsor: Alcoa
Program # 5
8. HYDROGEN DAMAGE MECHANISMS IN ADVANCED ALUMINUM ALLOYS**
Faculty Investigator: John R. Scully
Graduate Student: To be named
Department: Materials Science
NASA-LaRC Contact: W.B. Lisagor (Metallic Materials)
Start Date: January, 1991
Anticipated Completion Date: To be determined
Cosponsor: Virginia CIT

8. EFFECT OF ZINC ADDITIONS ON THE ENVIRONMENTAL STABILITY OF ALLOY 8090*

Faculty Investigator: Glenn E. Stoner
Graduate Student: Raymond J. Kilmer; PhD candidate
Department: Materials Science
NASA-LaRC Contact: W.B. Lisagor (Metallic Materials)
Start Date: September, 1989
Anticipated Completion Date: December, 1992
Cosponsor: Alcoa
Program # 6

9. DEFORMATION AND FRACTURE OF ALUMINUM-LITHIUM ALLOYS: THE EFFECT OF DISSOLVED HYDROGEN*

Faculty Investigator: R.E. Swanson (VPI)
Graduate Student: Frederic C. Rivet; MS candidate
VPI Department: Materials Engineering at VPI
NASA-LaRC Contact: D.L. Dicus (Metallic Materials)
Start Date: September, 1988
Completion Date: December, 1990
Program # 7

AEROSPACE MATERIALS SCIENCE

10. INVESTIGATION OF THE REACTION KINETICS BETWEEN SiC FIBERS AND SELECTIVELY ALLOYED TITANIUM MATRIX COMPOSITES AND DETERMINATION OF THEIR MECHANICAL PROPERTIES**

Faculty Investigator: F.E. Wawner
Graduate Student: Douglas B. Gundel; MS candidate
UVa Department: Materials Science
NASA-LaRC Contact: D.L. Dicus and W.B. Brewer (Metallic Materials)
Start Date: January, 1989
Anticipated Completion Date: December, 1990
Program # 8

11. **QUANTITATIVE CHARACTERIZATION OF THE SPATIAL DISTRIBUTION OF PARTICLES IN MATERIALS: APPLICATION TO MATERIALS PROCESSING***

Faculty Investigator: John A. Wert
Graduate Student: Joseph Parse; PhD candidate
UVa Department: Materials Science
NASA-LaRC Contact: D.R. Tenney (Materials Division)
Start Date: September, 1988
Anticipated Completion Date: December, 1991
Program # 9

12. **PROCESSING AND SPF PROPERTIES OF WELDALITE™ SHEET****

Faculty Investigator: John A. Wert
Graduate Student: To be named
UVa Department: Materials Science
NASA-LaRC Contact: T. Bayles (Metallic Materials)
Start Date: September, 1991
Anticipated Completion Date: To be determined

MECHANICS OF MATERIALS FOR AEROSPACE STRUCTURES

13. **INELASTIC RESPONSE OF METAL MATRIX COMPOSITES UNDER BIAXIAL LOADING***

Faculty Investigators: Carl T. Herakovich and Marek-Jerzy Pindera
Research Associate: Farshad Mirzadeh
Graduate Student: Mr. Clifford J. Lissenden, PhD Candidate
UVa Department: Civil Engineering
NASA-LaRC Contact: W.S. Johnson (Mechanics of Materials)
Start Date: September, 1990
Anticipated Completion Date: September, 1993
Program # 10

THERMAL GRADIENT STRUCTURES

14. DESIGN OF CRYOGENIC TANKS FOR SPACE VEHICLES*

Faculty Investigators: W.D. Pilkey and J.K. Haviland

Graduate Student: Charles Copper; PhD candidate

Undergraduate: Kara J. Peters

UVa Department: Mechanical and Aerospace Engineering

NASA-LaRC Contact: Drs. D.R. Rummeler (Structural Mechanics Division),
R.C. Davis and M.J. Stuart (Aircraft Structures)

Start Date: January, 1991

Anticipated Completion Date: December, 1991

Program # 11

15. EXPERIMENTAL STUDY OF THE NONLINEAR VISCOPLASTIC RESPONSE
OF HIGH TEMPERATURE STRUCTURES*

Faculty Investigator: Earl A. Thornton

Graduate Student: Marshall F. Coyle

UVa Department: Mechanical and Aerospace Engineering

NASA-LaRC Contact: James H. Starnes, Jr. (Aircraft Structures)

Start Date: January, 1990

Anticipated Completion Date: To be determined

Program # 12

* Progress is reported.

** Project will be initiated in January of 1991; progress is not reported here.

RESEARCH PROGRESS AND PLANS (July 1 to December 31, 1990)

Research progress, recorded during the period from January 1, 1990 to June 30, 1990 is summarized and future plans are described here for each of the eleven projects.

Program 1 **Environment Enhanced Fatigue of Advanced Aluminum Alloys and Composites**

Donald C. Slavik and Richard P. Gangloff

Objective

The broad objective of this PhD research is to characterize and understand the environmental fatigue crack propagation behavior of advanced Al-Li-Cu based alloys and metal matrix composites. Aqueous NaCl and water vapor, which produce atomic hydrogen by reactions on clean crack surfaces, are emphasized. The effects of environment sensitive crack closure, stress ratio and precipitate microstructure are assessed. We seek mechanistic models of intrinsic crack tip damage processes to enable predictions of cracking behavior outside of the data, metallurgical improvements in material cracking resistance, and insight on hydrogen compatibility.

The specific objective of work during this reporting period is to develop the experimental methods to achieve the above goals.

Introduction and Approach

The good fatigue crack propagation (FCP) resistance of wrought Al-Li-Cu alloys and metal matrix composites is traceable to beneficial extrinsic crack closure mechanisms and to reduced intrinsic hydrogen environment embrittlement^[1]. Such FCP behavior is well established for moist air, but is unexplored for advanced light alloys and composites in other aggressive gaseous and aqueous environments. The effects of alloy microstructure and stress

ratio on intrinsic hydrogen damage, and the contribution of environment sensitive closure mechanisms to FCP behavior are particularly uncertain. No quantitative model exists to predict crack growth rate from the hydrogen embrittlement perspective.

A previous study of environmental fatigue in alloy 2090 by Piascik, under LA²ST Program sponsorship, provides the foundation for this research^[2]. The conclusions from Piascik's work, remaining uncertainties, and the technical approach for Mr. Slavik's research were summarized in the recent proposal^[3]. Central to the proposed research is the need to develop the capabilities to measure the closure behavior of fatigue cracks in aluminum alloys in aggressive environments and to estimate the effect of crack surface contact on crack growth kinetics. Additionally, it is necessary to develop an experimental method to characterize the FCP behavior of limited amounts of advanced alloys and for well defined stress intensity and environmental conditions.

Progress During the Reporting Period

Specimen Design and K-Solution Verification

In the current research, fatigue crack growth rates (da/dN) and associated crack closure for advanced materials will be measured in various environments. Crack length is measured with the well established direct current potential difference procedure^[4]. The capability exists to conduct automated FCP experiments under programmed stress intensity control.

The fixed grip single edge notched (F-SEN) specimen is selected for study. This specimen is inexpensive to fabricate, is obtainable from thin sheets of advanced materials, and is effectively gripped. This specimen allows for variation in the magnitude of the far field bending stress by changes in the specimen width to grip separation distance. Stress states span the range from uniform uniaxial tension ("T stress" = 0) to bending domination (T stress > 0) typical of a compact tension or fully rotating SEN specimen. K-solutions for rigidly gripped SEN specimens were developed by Bowie, Freese, and Neal, and describe the effect of the specimen width to grip separation distance ratio on K ^[5].

Hydraulically actuated grips were delivered by Instron and installed on a

Intrinsic FCP Behavior of Al-Li-Cu Sheet Alloys in Moist Air

The F-SEN specimen, Bowie K-solution, programmed constant K_{\max} procedure and hydraulic grips were employed to characterize the intrinsic da/dN - ΔK behavior for two Al-Li-Cu alloys, Vintage III 2090 from Alcoa and 2091 (Mg addition) from Pechiney. Two L/W ratios (6.0 and 7.0) were evaluated because this grip separation is required to accommodate the environmental chamber. Each alloy was obtained as thin sheet and in the unrecrystallized T3 condition, although this vendor claim has not been confirmed. 2090 was peakaged at 190°C for 4 hours^[2]. T_1 (Al_2CuLi), δ' (Al_3Li), and θ' (Al_2Cu) are the predominate strengthening precipitates^[2]. δ' promotes localized planar slip which can lead to crack path tortuosity and a high degree of crack closure. Alloy 2091 was peakaged at 135°C for 12 hours^[6]. The predominate strengthening precipitate in peakaged 2091 is reported to be δ' (Al_3Li), with no evidence for T_1 or S' (Al_2CuMg)^[7]. F-SEN specimens were machined from aged sheet in the L-T orientation.

The intrinsic FCP behavior of 2090 and 2091 sheet is summarized in Figure 2. K_{\max} was maintained constant (17 MPa \sqrt{m}) for each alloy, as ΔK was continuously decreased. This loading sequence caused the stress ratio, R , to increase from 0.4 to above 0.9. Note the excellent agreement between the data for alloy 2091 from Figure 1 (solid circles with $L/W = 3.0$ and constant ΔK loading) and the results of the continuously decreasing ΔK experiment (open circles with $L/W = 6.0$). The crack growth response of each Al-Li alloy is equivalent over the full range of ΔK values. (L/W for 2090 equalled 7.0.) For each alloy, a complex da/dN - ΔK relationship is observed, as typified by different power-law regimes at high and low ΔK , with an intermediate ΔK transition regime and no clear evidence for a threshold at high R ΔK levels as low as 1.3 MPa \sqrt{m} . This complex da/dN - ΔK behavior is similar to that observed for plate alloy 2090, indicated by the crosses in Figure 2, and is solely due to the hydrogen environmental effect of moist air^[2,8].

The crack growth rate data, generated by the F-SEN procedure, agree with high stress ratio literature results for each alloy. The solid line in Figure 2 represents data for the same sheet of peak aged (T8; 135°C for 12 hours) 2091, but obtained with a freely rotating SEN geometry^[6]. The agreement with the current data (O) is good. The dashed line represents compact tension based results for peak aged (T8E41 or T83; 163°C for 24 hours) 2090 sheet,

- o Sheet forms of alloys 2090 and 2091 have equivalent intrinsic fatigue crack growth rates in moist air. Growth rates for 2090 sheet are enhanced over values for plate. The exact nature of this enhancement is not understood, but is likely due to a hydrogen embrittlement-microstructure interaction.
- o A crack closure gage for the fixed grip single edge notch specimen was developed and successfully indicated load crack mouth displacement behavior typical of crack closure for FCP in air.

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1. K.T. Venkateswara Rao, R.S. Piascik, R.P. Gangloff and R.O. Ritchie, "Fatigue Crack Propagation in Aluminum-Lithium Alloys", in Proc. Fifth Intl. Al-Li Conf., T.H. Sanders, Jr. and E.A. Starke, Jr., eds., Materials and Component Engineering Publications Ltd., Birmingham, UK, pp. 955-971 (1989).
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3. R.P. Gangloff, "NASA-UVa Light Aerospace Alloy and Structures Technology Program", UVa Proposal No. MS-NASA/LaRC-4841-91, University of Virginia, Charlottesville, VA (1990).
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5. O.L. Bowie, C.E. Freese, and D.M. Neal, "Solution of Plane Problems of Elasticity Utilizing Partitioning Concepts", Trans. ASME J. of Applied Mechanics, pp. 767-772 (1973).
6. P. Coppin and R.P. Gangloff, "Behavior of Short Fatigue Cracks in Aluminum-Lithium Alloys", Unpublished Research (1989).
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10. R.J. Rioja, private communication, ALCOA Laboratories, Alcoa Center, PA (1990).
11. K.T. Venkateswara Rao, W. Yu, and R.O. Ritchie, "Fatigue Crack Propagation in Aluminum-Lithium Alloy 2090: Part I. Long Crack Behavior, Part II. Small Crack Behavior", Metallurgical Trans. A, Vol. 19A, pp. 549-569 (1988).

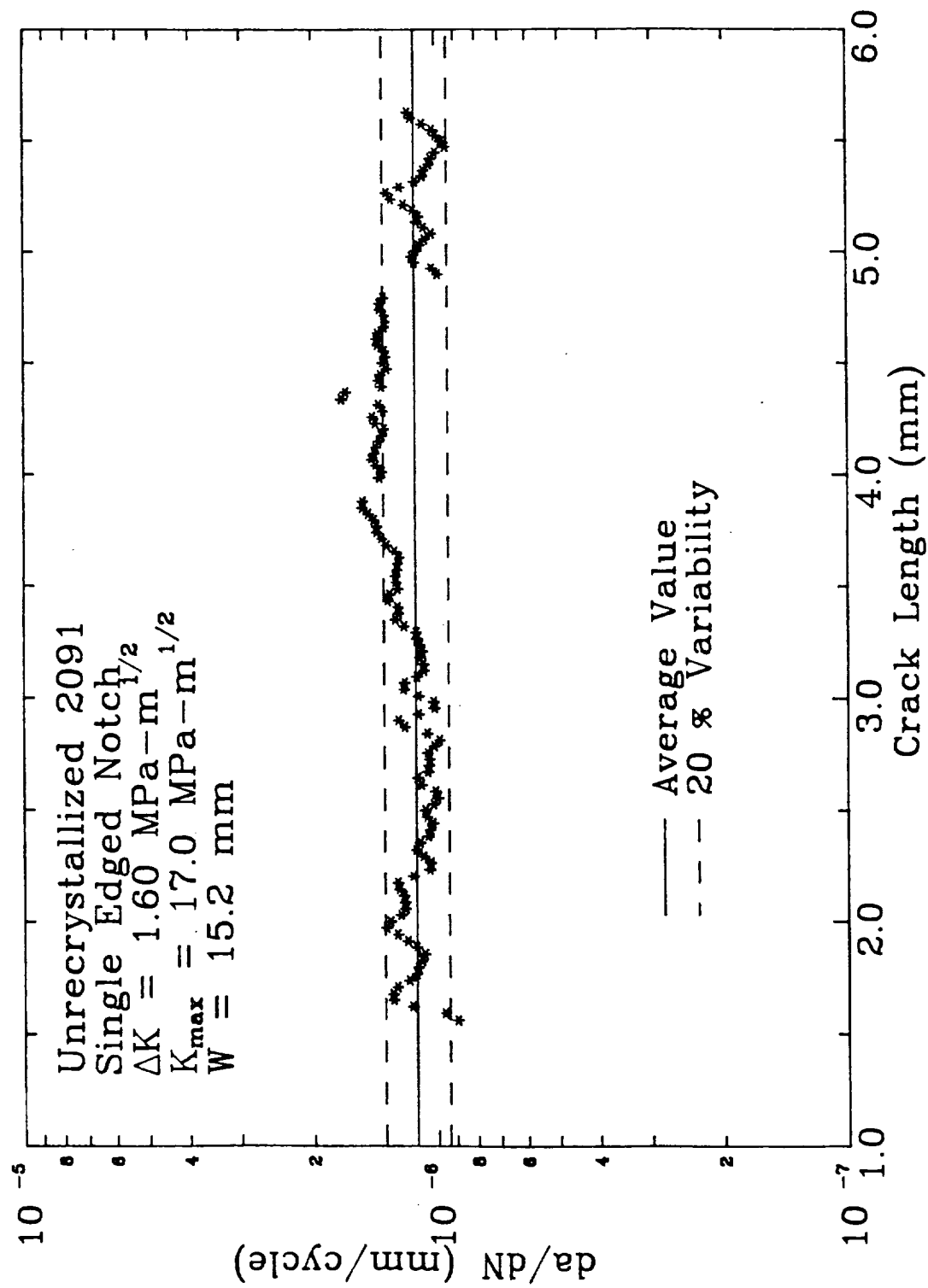


Figure 1 The experimental K-solution verification for the fixed grip SEN geometry ($L/W = 3$).

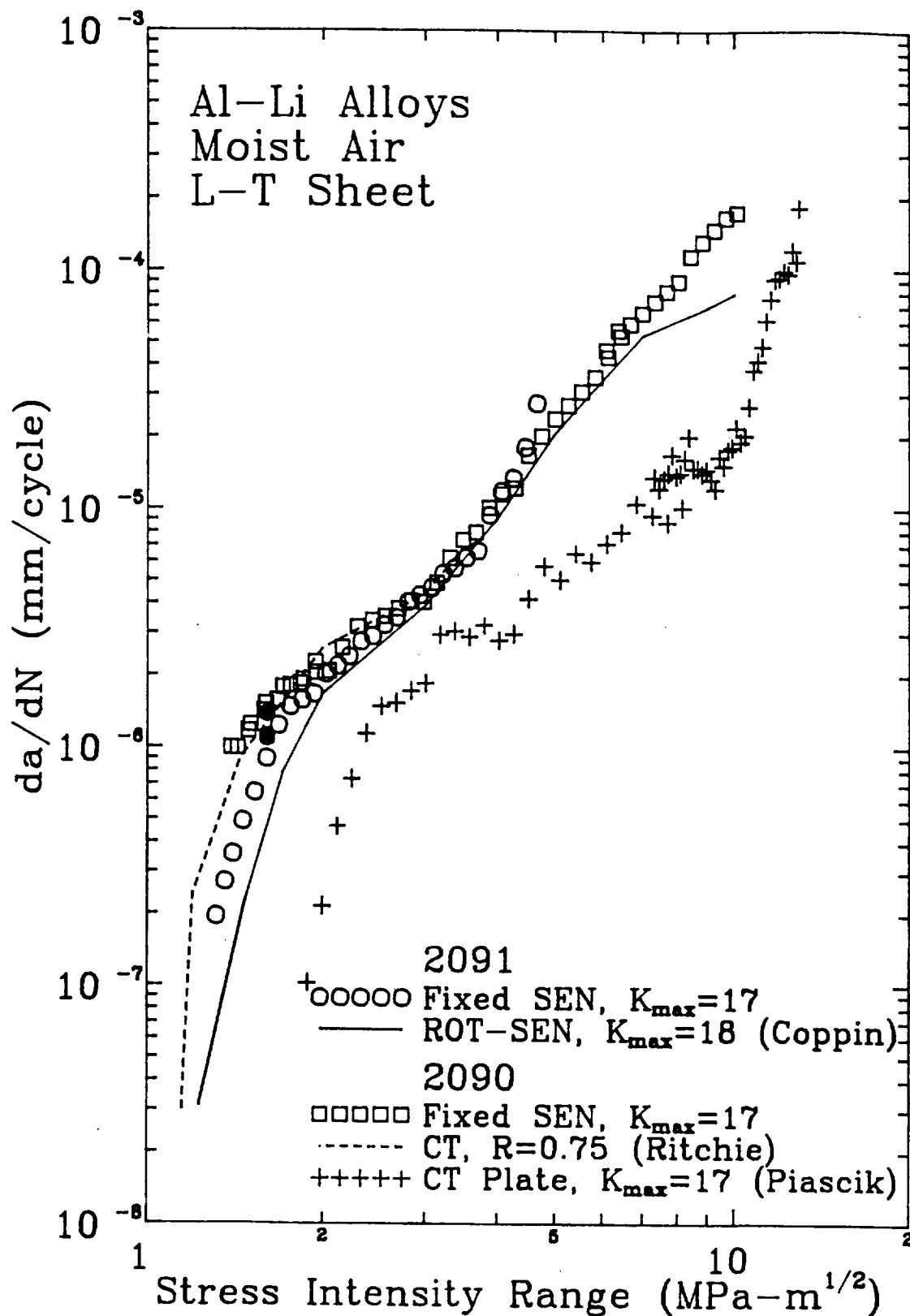


Figure 2 The intrinsic fatigue crack growth rate behavior of Al-Li-X alloys 2090 and 2091 in moist air.

Program 2 Elevated Temperature Fracture of an Advanced Powder Metallurgy Aluminum Alloy

William C. Porr, Jr. and Richard P. Gangloff

Objective

The goal of this PhD research is to characterize the fracture behavior of advanced powder metallurgy Al-Fe-V-Si alloy 8009 (previously called FVS0812) under monotonic loading, as a function of temperature. Particular attention is focused on contributions to the fracture mechanism from the novel fine grained dispersoid strengthened microstructure, dissolved solute from rapid solidification, and the moist air environment.

Approach

The program approach and experimental procedures were outlined in previous reports and presentations^[1-4]. Work prior to this current reporting period has focused on the effect of temperature on the tensile and fracture toughness properties of rapidly solidified PM 8009 and ingot metallurgy alloy 2618. Fracture behavior was quantitatively characterized by initiation and growth fracture toughnesses and J-integral fracture mechanics, and qualitatively in terms of microscopic fracture processes. Subsequent experiments were planned to examine mechanistic questions raised by the unique fracture behavior exhibited by 8009.

Work over the past six month period includes microscopy to examine prior particle boundary delamination, evaluation of fracture properties as a function of loading rate at 175°C, qualification of a second extrusion of 8009, and initial study of the effect of cross-rolling on fracture behavior. Results of the current study were presented and published at the 1990 TMS Fall Meeting in Detroit^[5], and the academic requirement of a PhD dissertation proposal presentation was completed.

Research Progress and Results

Microscopy of Prior Particle Boundary Delamination

Delamination toughening, described in previous reports^[3-5], may be an important mechanism in the fracture behavior of 8009. Briefly, delamination of prior particle boundaries in the through thickness of an LT oriented fracture mechanics specimen causes an increase in the critical stress intensity for fracture, due to the loss in through-thickness constraint. Low magnification fractography has shown that delamination is present in 8009 when monotonically loaded to failure at 25°C^[3-5], however, there was no proof that it occurred prior to initiation of crack growth from the fatigue precrack. For delamination to affect initiation toughness (K_{IC}), it must occur prior to initiation.

To verify that delamination was indeed occurring prior to initiation, an LT oriented compact tension (CT) specimen was loaded to a stress intensity of 34.1 MPa/m (approximately K_{IC}) and then sectioned for microscopy. The LT oriented compact tension specimen was metallographically sectioned on two separate planes: (a) ahead of and perpendicular to the precrack front, parallel to the rolling-through thickness plane (perpendicular), and (b) parallel to the rolling-transverse plane, at mid-thickness and including the fatigue precrack (profile). Each section was mounted and electrolytically polished. Figure 1 shows the profile view; considering the precrack tip, it is evident that K_{IC} was exceeded and 0.1 mm of crack growth occurred. Since K_{IC} was exceeded, it cannot yet be proven that delamination occurred prior to crack initiation, however, it is shown in both Figs. 1 and 2 that delamination occurs ahead of the crack tip.

The profile plane in Fig. 1 was polished at a slight angle to the rolling plane and allows an estimation of how far ahead of the crack tip delaminations extend. It may be significant that this distance is on the order of the plane strain crack tip plastic zone size (300 μ m) for this material. Figure 2 is the sectioned specimen ahead of the crack tip and shows that the delaminations follow along the prior particle boundaries perpendicular to the crack front.

Figure 3 is a metallographic cross section of a fractured CT specimen that shows the depth that delaminations extend below the crack surface (top). (This polished plane is

perpendicular to the transverse direction of crack growth and is parallel to the longitudinal-transverse plane for the LT oriented specimen.) Several delaminations are evident, parallel to the longitudinal plane of the extrusion. "Necking" of failed ligaments between delaminations, from a loss of through-thickness constraint and the resulting plane stress shear deformation, is apparent in this micrograph and is evidence that delamination occurs before crack growth in the ligament. The depth of the delamination, below the main fracture surface, is on the order of the plane strain plastic zone size, as necessary for relaxation of constraint.

Effect of Loading Rate

Table I and Fig. 4 give the results of experiments on the effect of actuator displacement rate on initiation and growth fracture toughnesses (K_{IC} and T , respectively) of AA 8009 at 175°C. Both K_{IC} and T decrease with decreasing loading rate. This decrease is sharp (K_{IC} : 23.2 ± 1.9 to 14.2 ± 1.1 MPa/m, T : 6.1 to 2.6) with the initial rate decrease from 2.54×10^{-2} to 2.54×10^{-3} mm/s¹. Further toughness decreases are mild. K_{IC} may in fact be rate independent and the tearing modulus exhibits a minimum of 1.4 at an actuator displacement rate of 2.54×10^{-5} mm/s. It is uncertain if the relatively high toughness at 1.02×10^{-5} mm/s represents the real behavior of this material or an aberrant datum point.

Increases of T with lower loading rates correspond to the observation of prior particle boundary delamination and probable delamination toughening as discussed in previous reports^[3-7]. Delamination was not present in the specimens tested at loading rates higher than 1.02×10^{-5} mm/s. This observed sensitivity of T , but not K_{IC} , to delamination implies that delaminations occur after the initiation of crack growth; that is at stress intensity levels above K_{IC} . The fact that a large decrease in toughness occurred as loading rate decreased from 2.54×10^{-2} to 2.54×10^{-3} mm/s, despite the absence of delamination in both cases, implies that some time dependent intrinsic embrittlement mechanism is active. This result is critical to

¹The interval plotted with each datum point in Fig. 4 and Table 1 represents the uncertainty associated with the definition of the onset of crack propagation, as inferred from measured electrical potential. This uncertainty is due to system noise and thermally induced voltage variations, and to possible crack opening and plastic strain effects on the potential.

mechanistic understanding of the fracture behavior of rapidly solidified PM aluminum alloys.

Table I. Initiation and Growth Fracture Toughnesses of AA 8009 at 175°C as a Function of Loading Rate

Displacement Rate (mm/s)	K _{IC} (MPa√m)	T
2.54x10 ⁻²	23.2 ± 1.9	6.1
2.54x10 ⁻³	14.2 ± 1.1	2.6
2.54x10 ⁻⁴	12.5 ± 1.8	1.5
2.54x10 ⁻⁵	11.7 ± 0.7	1.4
1.02x10 ⁻⁵	13.4 ± 1.5	3.2
5.08x10 ⁻⁶	11.1 ± 0.3	2.7

The "embrittlement" of 8009 with decreasing loading rate is consistent with the results of sustained-load crack growth and frequency-dependent elevated temperature fatigue studies^[8,9]. The same intrinsic mechanisms proposed for the toughness behavior of this alloy as a function of temperature may explain these loading rate effects^[3-7]. These mechanisms include dynamic strain aging, hydrogen embrittlement from the moist air test environment and/or retained hydrogen from processing, and localized plastic deformation and microvoiding.

The time dependence of delamination exhibited here can be explained by a time dependent decrease in prior particle boundary strength. No micromechanism for this decrease is proposed at this time.

Qualification of Second Extrusion

A second extrusion of 8009 was donated by Allied-Signal, Inc. It was desired to confirm the uniformity of properties from one extrusion to the next, so as to not introduce uncertainty to subsequent experiments. It is additionally important to assess the generality

of the fracture behavior of 8009.

The results of fracture toughness experiments as a function of temperature are given in Table II. The second extrusion had similar fracture toughnesses compared to the first at all temperatures. Macroscopically, the fracture surfaces of the second also were similar to those of the first. Delamination was evident in specimens tested at the standard high loading rate at 25°C and 316°C, but was absent in the CT specimen fractured at 175°C. Additional fractography is planned along with examination of tensile properties.

Table II. Initiation and Growth Fracture Toughnesses of AA 8009 as a Function of Temperature.

Temperature (°C)	Extrusion	K _{IC} (MPa/m) ^a	T ^a
25	1	34.6 ± 8.7	22.9
25	1	35.6 ± 4.5	16.4
25	2	34.9 ± 4.3	25.9
25	2	30.5 ± 3.6	--- ^b
100	1	27.7 ± 1.9	6.8
175	1	14.2 ± 1.1	2.6
175	2	15.8 ± 0.6	2.2
200	1	15.5 ± 1.0	2.2
225	1	15.3 ± 0.4	4.7
316	1	10.5 ± 3.2	12.8
316	1	10.6 ± 0.5	12.9
316	1	10.8 ± 2.0	12.7
316	2	9.6 ± 0.6	11.9

^a Actuator Displacement Rate = 2.54x10⁻³ mm/s

^b Insufficient stable crack growth to determine T

Based on the fracture toughness results, the newly received extrusion appears to be similar to the first, allowing direct comparison of future experiments with those already completed. The effect of temperature on fracture toughness is general.

Cross-Rolled Plate

Alloy 8009 in the form of 0.25" cross-rolled plate was obtained from the Metallic Materials Branch of the NASA-Langley Research Center. It was speculated that rolling (90° to the extrusion direction) would break up the prior particle boundaries in the 8009 and result in an isotropic fracture behavior. Such processing should greatly reduce delamination for the LT orientation and perhaps improve the fracture toughness for the TL orientation. Metallography showed that the oxides on prior ribbon particle boundaries were more diffuse than in the extruded product.

Table III and Fig. 5 show the results of preliminary fracture toughness testing as a function of temperature with plate 8009. Initiation and growth fracture toughnesses are independent of specimen orientation (with respect to the rolling direction). No delamination was observed in the specimens tested at either 25°C or 175°C, however, delamination was present in the specimen fractured at 316°C. Initiation toughness values for the plate are approximately 15 to 20% lower than in the extruded product at all temperatures, raising questions as to the magnitude of the influence of delamination on K_{IC} for the extrusion.

For 23°C, the tearing modulus for plate 8009 was significantly less than that of the extrusion, due to delamination in the latter case. In Tables II and III, stable crack growth was extensive for three of the four specimens from the extrusions, but was limited for all plate specimens. In one of the extrusion specimens and in each plate specimen, some stable crack growth occurred (T is greater than 0), however, the amount was less than that needed for our standard definition of T . Physically, this indicates reduced crack growth resistance, however, additional analysis is required to quantify this effect.

Without the complication of delamination in specimens of the plate 8009, tested from 25 to 175°C, the decrease in K_{IC} with increasing temperature is proof of a significant intrinsic embrittlement phenomenon associated with this RSR PM alloy. The substantial rise in tearing modulus at 316°C corresponds to the occurrence of delamination, again illustrating

the sensitivity of T to delamination.

Table III. Initiation and Growth Fracture Toughnesses of Cross-Rolled AA 8009 Plate a Function of Temperature

Temperature (°C)	Specimen Orientation	K _{IC} (MPa/m) ^a	T ^a
25	LT	29.2 ± 2.5	--- ^b
25	LT	29.0 ± 2.6	--- ^b
25	TL	28.8 ± 3.0	0
175	LT	9.9 ± 0.2	2.5
175	TL	10.0 ± 0.7	2.2
316	LT	8.2 ± 0.6	13.0

^a Actuator Displacement Rate = 2.54x10⁻³ mm/s.

^b Insufficient stable crack growth to determine T

Conclusions

1. Fractography shows that delamination occurs along oxide decorated prior particle boundaries, ahead of the crack tip, in monotonically loaded CT specimens of AA 8009. Necking of failed ligaments between delaminations is proof that through thickness constraint relaxes prior to crack extension.
2. Initiation and growth fracture toughnesses of AA8009 decrease with decreasing loading rate at 175°C due to an undetermined time dependent embrittlement phenomenon. Crack growth fracture toughness (T) exhibits a minimum before increasing at the lowest loading rates due to the advent of through-thickness delamination and subsequent toughening.

3. A newly acquired extrusion of 8009 has the same temperature dependent fracture toughness properties as a previously tested extrusion.
4. Experimentation with cross-rolled 8009 plate verifies a significant embrittlement phenomenon at elevated temperatures. The fracture toughness properties of the plate are independent of specimen orientation (LT and TL) because the rolling process altered the oxide decorated prior particle boundaries.

Plans for Future Work

A detailed description of proposed research is given elsewhere^[6,7]. Work for the next reporting period includes continuation of experiments with the cross-rolled plate, fracture toughness testing of extruded 8009 in vacuum, and tensile experiments with notched specimens.

The experiments in vacuum are intended to explore the intrinsic embrittlement phenomenon in 8009 by eliminating possible hydrogen uptake from the moist air environment at elevated temperatures. The vacuum chamber and servohydraulic loading system in the Environmental Fracture Laboratory at UVA have been modified for elevated temperature testing up to 200°C with better than $\pm 1^\circ\text{C}$ stability and at a vacuum of better than 10^{-7} torr.

Experiments with the notched tensile bars will allow examination of the effect of constraint on the microscopic fracture processes in 8009. Additionally, the design of these specimens will facilitate sectioning necessary for microscopic study of the evolution of damage in the ultra-fine grained 8009. A diametral extensometer has been purchased from Interlaken Technology Corp. to monitor notch root strain in the specimens.

References

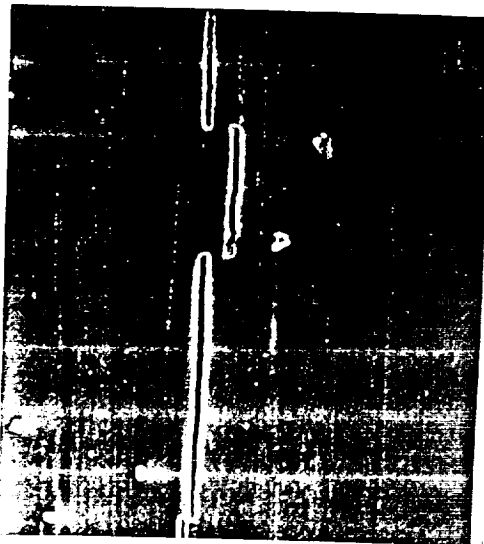
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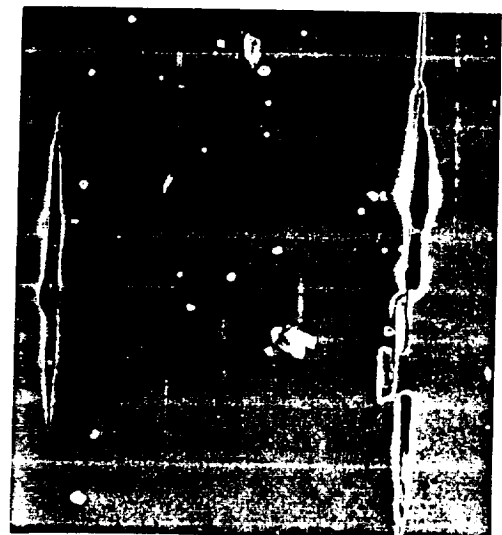
100 μ m

Figure 1. Profile view of an AA 8009 CT specimen with delamination along oxide decorated prior particle boundary ahead of the crack tip.



(a)

100 μ m



(b)

100 μ m

Figure 2. Sections of an AA 8009 CT specimen, (a) and (b), ahead of the crack tip with delaminations perpendicular to the crack front.

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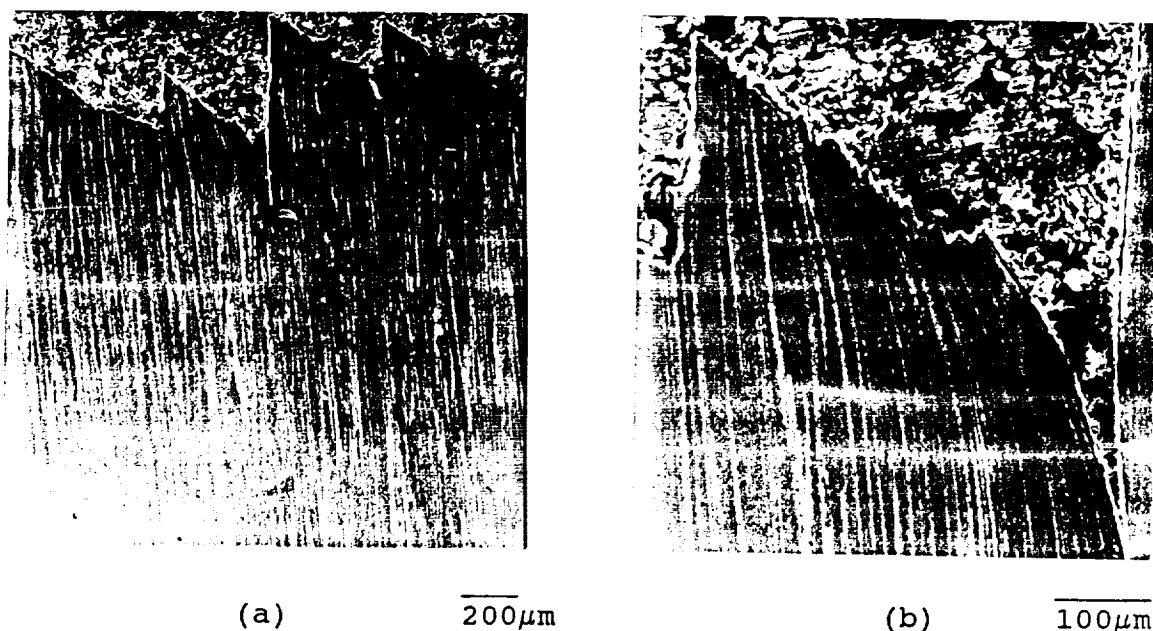


Figure 3. Cross section of AA 8009 CT specimen fracture surface indicating ligament necking in between delaminations.

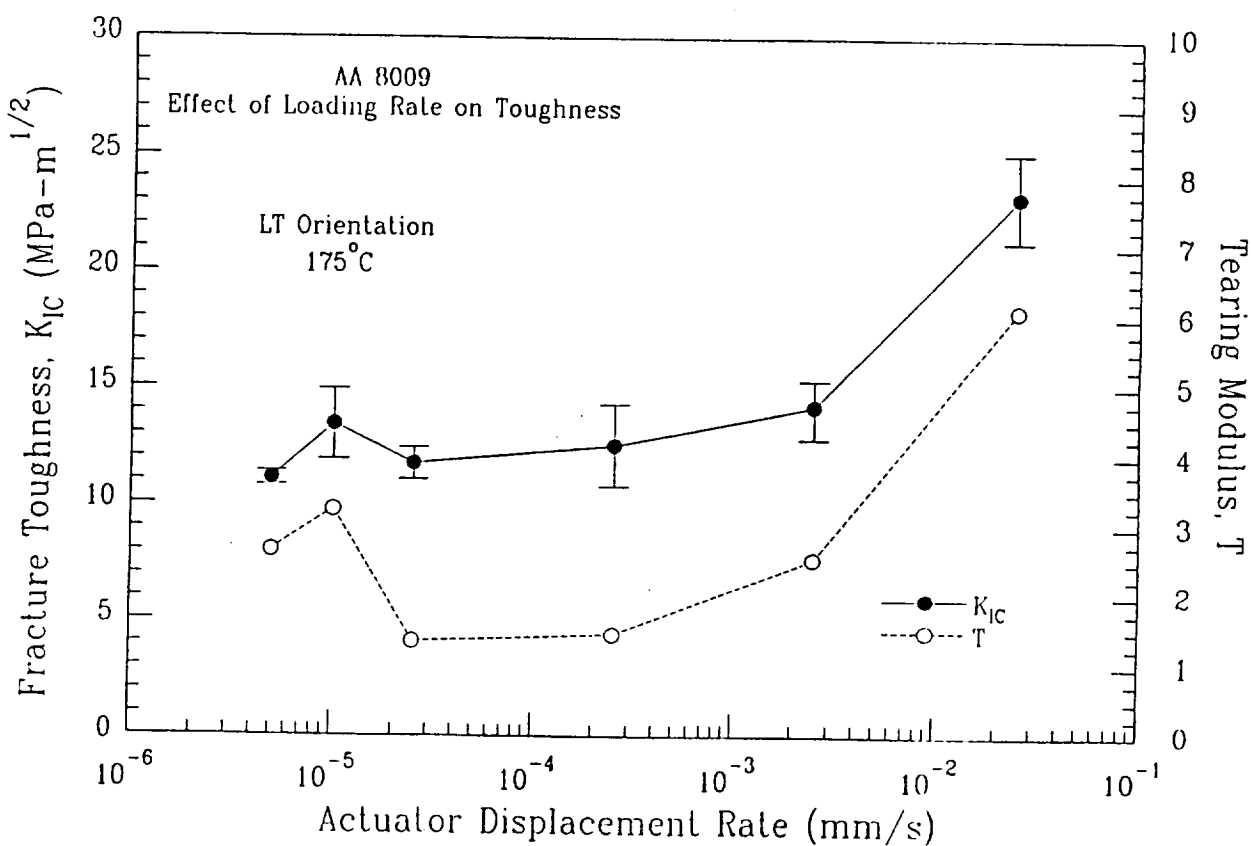


Figure 4. Initiation (K_{IC}) and growth (T) fracture toughnesses as functions of actuator displacement rate for extruded AA 8009. LT orientation. Test temperature = 175°C.

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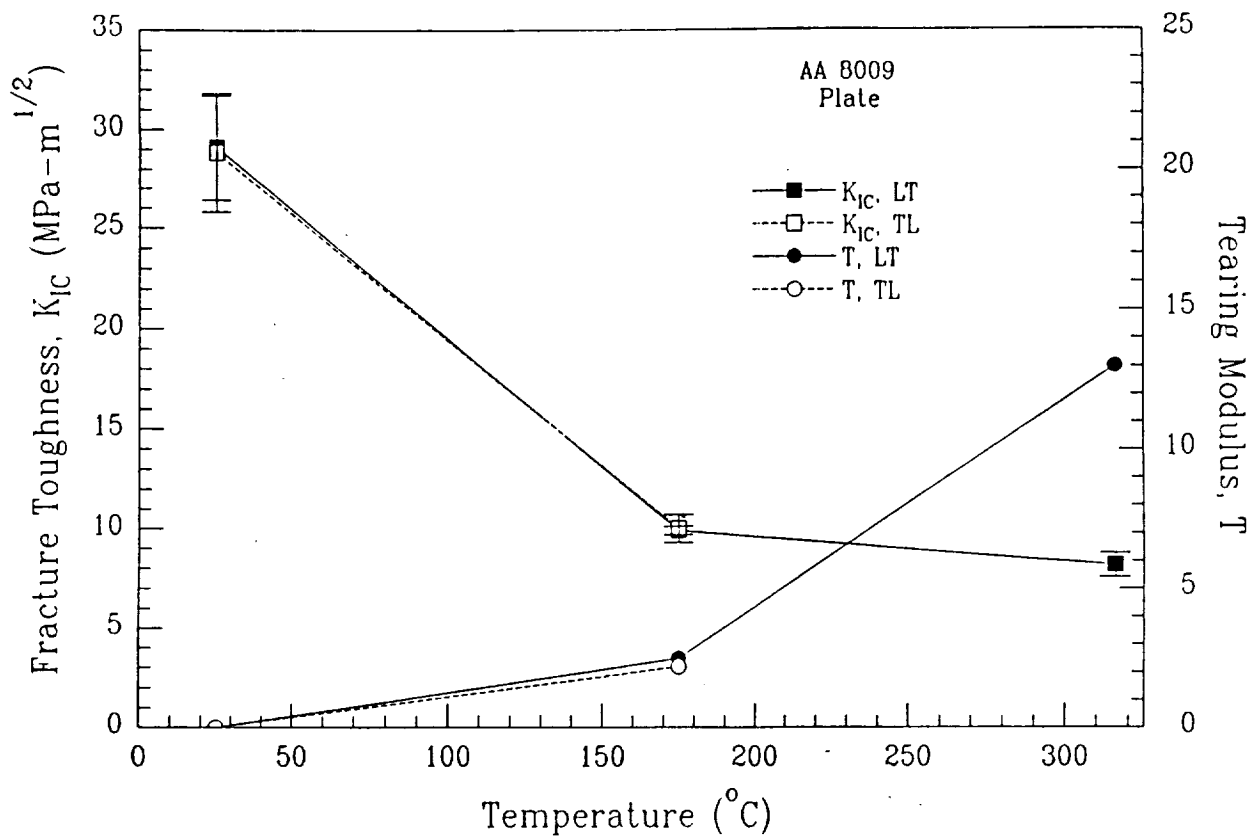


Figure 5. Initiation (K_{IC}) and growth (T) fracture toughnesses as functions of temperature and specimen orientation for cross-rolled AA 8009 plate. Actuator displacement rate = 2.54×10^{-3} mm/s.

Program 2 Elevated Temperature Crack Growth in Advanced Aluminum Alloys
Yang Leng and Richard P. Gangloff

Objective

The objective of this study is to characterize time-dependent crack growth (TCG) in advanced aluminum alloys at elevated temperatures with the fracture mechanics approach, and to examine TCG mechanisms with a metallurgical approach. Specific tasks in the past six months were:

- * to obtain TCG experimental information from a refined testing system.
- * to correlate TCG with the J-integral.
- * to investigate the intermediate temperature embrittlement of 8009 alloy in order to understand TCG mechanisms.

Summary

With refined test and data acquisition systems, subcritical crack growth was observed at lower stress intensity levels than that in previous experiments. At 175°C crack growth was observed at 40% of the load at which the "fast loading rate" K_{IC} is defined. The J-integral correlates TCG rates in 8009 (previously called FVS0812) and the general correlation with crack growth rate is similar to that provided by K. The influence of J due to plastic deformation is evident at higher loads and temperatures. Refined tensile tests for 8009 at 175°C did not show serrated stress-strain curves, as common to dynamic strain aging. Analytical and experimental estimations indicated that Fe and V solute contents in 8009 are, at least, 10 times higher than the equilibrium solid solubilities in aluminum. Such high solute contents may play a role in intermediate temperature embrittlement of this alloy.

Background

A previous report indicated that time dependent crack growth occurs in ingot metallurgy 2618 and in powder metallurgy (PM) 8009 alloys^[1]. Subcritical crack growth occurs at elevated temperatures and at stress intensity levels below the critical value for crack initiation (K_{IC}). Also, the previous study established that the creep crack growth parameters, C^* -integral and C_I , are not valid parameters to correlate TCG in these aluminum alloys because there is limited creep deformation during crack growth. In other words these aluminum alloys are creep brittle. Correlation between the crack growth rate and the elastic stress intensity, K , was found^[1].

The J-integral is a nonlinear fracture mechanics parameter which represents the magnitude of the stress-strain field (HRR field) at the crack tip. At sufficiently high proportions of the fully plastic limit load (e.g., large a/W , high applied loads or high temperatures), the effect of instantaneous plastic deformation during TCG cannot be ignored. In this sense the J-integral may be a better parameter to correlate crack growth at elevated temperatures because it can account for both the elastic and plastic driving forces.

Commonly, there are two methods to determine the J-integral: the estimation procedure and the area method. The estimation method, given in the Electric Power Research Institute handbook^[2], estimates the J-integral based on finite element results for a hardening material. In order to use this method, the stress state in the crack tip region (plane stress or plane strain) and the power law plastic stress-strain relation must be known. Unfortunately, stress state is uncertain for laboratory specimens and this problem is exacerbated for low work hardening alloys such as 2618 and 8009 at elevated temperatures^[1,3]. The area method is commonly employed to determine applied J-integral levels for bending dominated test pieces.

Conventional tests for J determination are conducted under the conditions of constant displacement rate, i.e. the testing system is in actuator or crosshead displacement control. Since the TCG tests are conducted at constant load, the validity of using conventional techniques and formulations for the J-integral determination needs to be verified. Before our investigation, this question had not been addressed in the literature. The first task was, therefore, to theoretically verify the area method for constant load crack growth, then to

apply the correct method to correlate J with TCG.

The success of elevated temperature crack growth tests depends on the thermal stability and sensitivity of the instrumentation for maintaining constant temperature and for measuring load, crack length and crack opening displacement. The thermal stability of the load cell, the level of electronic noise and long term stability of direct current potential drop signals, and LVDT calibrations are critical factors. We have optimized our system in these regards during this reporting period.

It was found in a previous study that the fastest rate of subcritical crack growth in alloy 8009 is at 175°C^[1]. This is coincident with the ductility and fracture toughness minima observed for this alloy^[4]. A possible explanation for temperature dependent embrittlement is dynamic strain aging (DSA). Microscopically, DSA is explained by solute atom migration with and locking of otherwise mobile dislocations. DSA is commonly characterized by a serrated stress-strain response and by inhomogeneous slip markings on the surfaces of tensile specimens^[5]. For PM 8009 strengthened with ultra-fine grain and a high volume fraction of dispersoids, however, the DSA features remain unknown.

Results

Elevated temperature crack growth experiments were conducted with alloy 8009 and with a refined testing system. In the refined testing system, a constant temperature cooling machine was used to control load cell temperature by circulating an alcohol-water mixture which is initially at 10°C. An improved data acquisition program was used to obtain more accurate potential difference signals by averaging more than 300 readings for each data point and by employing a new algorithm for accounting for spurious thermally induced voltages.

The new test results indicate that subcritical crack growth occurs in 8009 at lower stress intensity levels than preliminary tests, as shown in Fig. 1. Time-dependent crack growth can commence at 40% of the stress intensity at which the "fast loading rate" K_{IC} is defined when 8009 is tested at 175°C. Experiments at lower stress intensities did not indicate crack growth for loading times up to 50 hours. For such cases, variations in the electrical potential signal obscure very low rate crack propagation which may actually occur.

In order to understand the mechanism for TCG in precipitation strengthened aluminum alloys, crack growth and tensile creep tests were conducted with alloy 2618 at 150 to 200°C. The analysis of these experiments is currently progressing.

To analytically verify the area method for J-integral determination under constant load conditions, we started from the basic formulation of the J-integral as given by either of the following equivalent formulations:

$$J = -\frac{1}{B} \int_0^V \left(\frac{\partial P}{\partial a} \right)_V dV \quad (1)$$

$$J = \frac{1}{B} \int_0^P \left(\frac{\partial V}{\partial a} \right)_P dP \quad (2)$$

where P is applied load, V is load line displacement, a is crack length and B is specimen thickness. Equation 1 has been used to derive the J-integral in terms of an area associated with measured P-V data for constant displacement rate loading where load changes with crack length and dP/da is determined at any constant V. Considering constant load, we derived the P-V area expression for J from Eq. 2. This analysis shows that the identical J-integral formulation can be obtained from Eq. 1 or Eq. 2, assuming a power-law relation between load and load line displacement. The conventional formulation of J based on the P-V area method is valid for constant load tests.

Figure 2 shows the correlation between the crack growth rate and the J-integral which is determined by the area method defined by ASTM standard E813-87. J correlates sustained load crack growth rate; the trend of da/dt increasing according to a power-law with J, above a possible threshold, is similar to the trend of rate increasing with K because the elastic portion of J is dominant. The influence of the plastic portion of J is evident at higher loads and temperatures where the crack growth trends as a function of temperature differ when correlated with J and K. Since J incorporates ligament plastic deformation, if present, it is a proper parameter to represent the crack driving force.

In order to understand intermediate temperature embrittlement in 8009, a carefully designed tensile test was conducted. A cylindrical tensile specimen was polished to a nearly mirror finish and was strained at a tensile displacement rate of 0.5 mm per minute at 175°C. No slip markings were observed on the specimen surface and the stress-strain curve provided no indication of serrations. This implies that, if DSA occurs for alloy 8009, the features are different in this ultra-fine grain dispersoid strengthened PM alloy compared to conventional aluminum alloys.

The Fe and V solute contents in the 8009 aluminum matrix phase were analytically and experimentally estimated. Assuming that the volume fraction of the dispersoids is 24%, the total Fe and V contents in the matrix were calculated as 0.5 to 0.7 at %. The total Fe and V solute contents, measured from EDX analysis with the STEM, are in the range of 0.4 to 0.6 at %. This result is consistent with that from the calculation. Since the equilibrium solid solubility of Fe in aluminum is only about 0.03 at %, this indicates that rapid solidification results in metastable high concentrations of Fe and V solute atoms which could play a role in the intermediate temperature embrittlement. This conclusion is speculative, however, because accurate measurements of trace element contents in STEM are difficult.

Conclusions

1. Subcritical crack growth in alloy 8009 occurs at low stress intensities.
2. The J-integral correlates TCG in 8009; the general correlation with crack growth rate is similar to that provided by K.
3. If DSA exists in alloy 8009, the features and mechanism are different from conventional aluminum alloys.

Further work

We will continue to improve the TCG testing system by employing a dead weight testing frame to eliminate thermally induced load changes typical of servohydraulic or

servoelectric equipment. We will finalize the characterization of the time-dependent creep crack growth kinetics for 8009 and 2618, and will model macromechanical behavior with microstructural parameters. The mechanism study will include three aspects of damage leading to subcritical crack growth: localized creep deformation, microstructural degradation, and environmental attack. We will conduct creep crack growth experiments in the high vacuum system to eliminate the contribution from the external environment. This fundamental mechanism study will help to understand subcritical crack growth at elevated temperatures in creep brittle materials.

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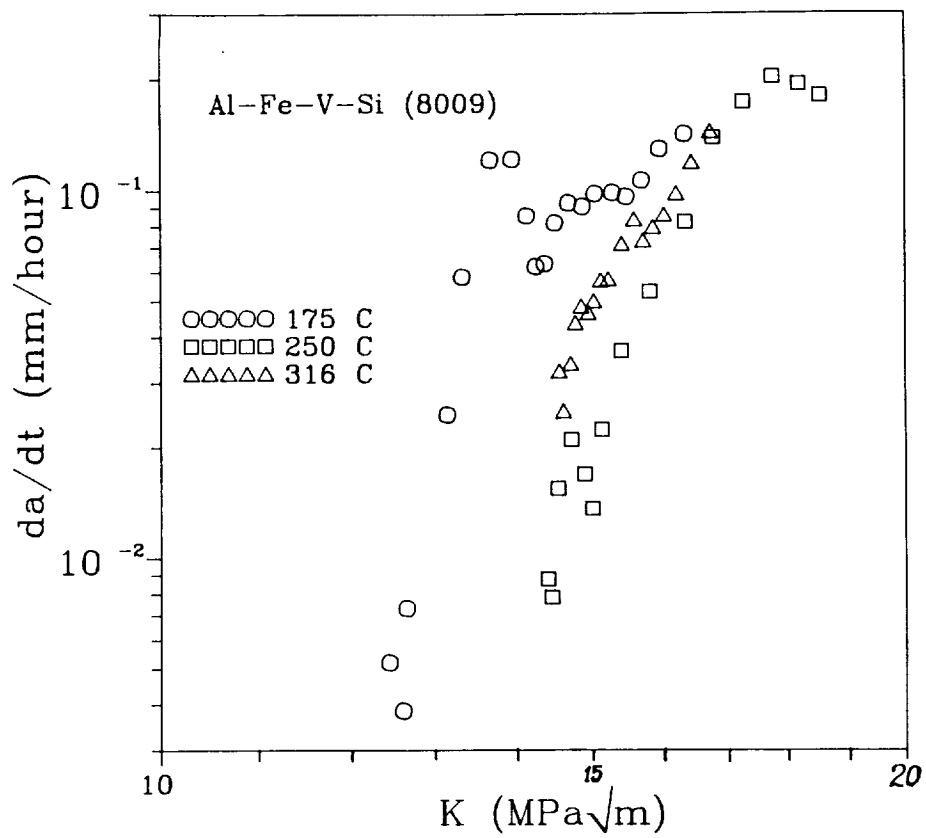


Fig. 1 Subcritical crack growth rate as a function of K.

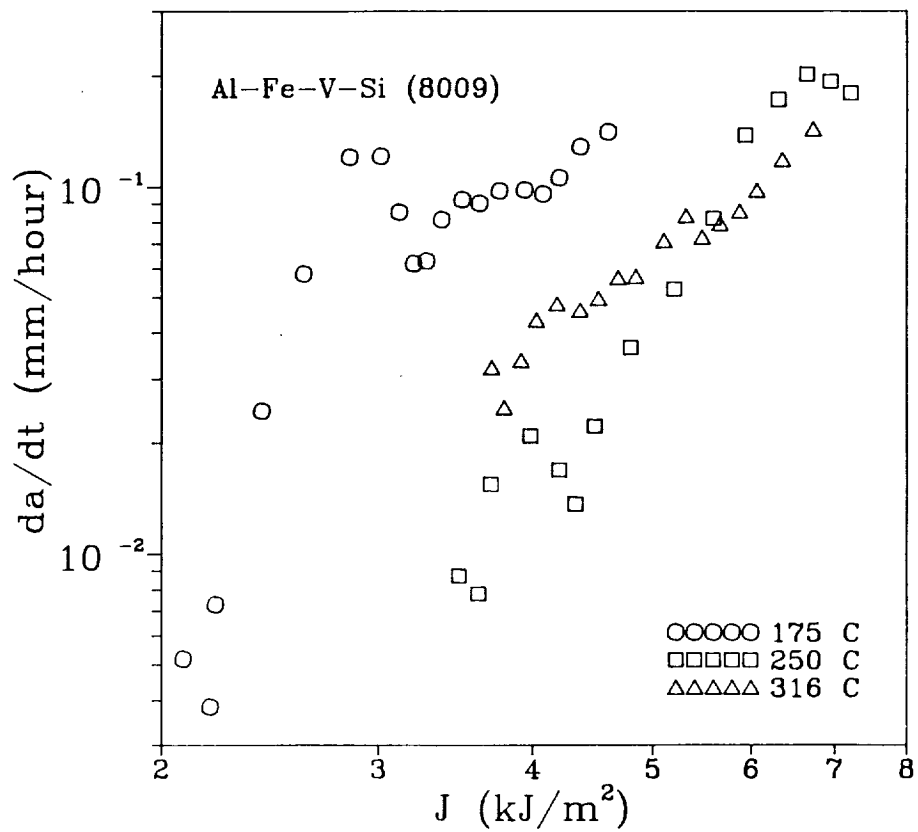


Fig. 2 Subcritical crack growth rate as a function of J.

Program 3 **Temperature Effects on the Deformation and Fracture of Al-Li-Cu-In Alloys**

John A. Wagner and R.P. Gangloff

Objective

The objective of this PhD research is to characterize and optimize the crack initiation and growth fracture resistance of Al-Cu-Li and Al-Cu-Li-In alloys for cryogenic tank applications. The program aims to understand microscopic fracture mechanisms, as influenced by ambient to cryogenic temperature, stress state and microstructure.

Introduction

Cryogenic propellant tanks account for a major portion of the structural weight of current and proposed launch vehicles. Significant weight and system costs savings could be realized in future launch vehicles by using innovative forming and joining processes and high strength, low density Al-Li alloys.^[1]

The results of initial Kahn tear fracture toughness experiments on sheet thickness Al-Cu-Li and Al-Cu-Li-In alloys were previously published^[2]. Based on these results, the background and approach to future PhD research was presented by Mr. Wagner in a proposal to his Doctoral Committee^[3]. Presently, this work is concentrating on determining the effects of stress state and temperature on the fracture toughness of 2090 and indium modified 2090 alloys. During this past year, precracked specimen J-integral fracture toughness tests were conducted on Al-Cu-Li and Al-Cu-Li-In alloys at ambient temperatures and in the plane stress and plane strain conditions. This approach characterizes both the crack initiation toughness, J_{IC} and K_{IC} , and the resistance to stable crack growth under plane stress deformation, given by the tearing modules.

Results for the Reporting Period

During this reporting period significant progress has been made in the experimental

work for Mr. Wagner's PhD research. These technical accomplishments are summarized, with major conclusions presented in italics.

Alloy Strength: Indium additions to an Al-Cu-Li 2090 composition increase ultimate tensile strength compared to that of 2090-T8, however, In does not effect yield strength. Two alloys were emphasized during this reporting period. Vintage III 2090-T81 is commercially available from Alcoa and is being used as a baseline for comparing experimental Al-Li-Cu-In alloys. The alloy was received in the form of 19.0 mm plate which was solution treated, stretched and peak aged (163°C for 24 hours) by Alcoa. This alloy, designated as A2090-T81, exhibits excellent tensile properties, Figure 1. The indium modification of 2090 was obtained in the form of T4 plate from a large scale casting by Reynolds Metals, as described in previous reports. After solution treatment by Reynolds, this material (R2090+In-T6) was aged at 160°C for 75 hours at NASA-LaRC to produce essentially maximum hardness, as previously established. Tensile strengths are indicated in Figure 1.

The data in Figure 1 and previous results with sheet material^[2] indicate that In is similar to the T8 stretch with regard to increasing hardness and ultimate tensile strength compared to 2090-T6. The lack of an In effect on yield strength is not understood. Alloy texture and microstructure were examined during this reporting period to better define the effect of indium.

Alloy Microstructure: Texture analysis revealed that the microstructures of A2090-T81 and R2090+In-T6 were primarily unrecrystallized and typical of commercially available Al-Li-Cu-Zr plate. Full {100} and {111} pole figures were determined at the Alcoa Technical Center for A2090-T81 and R2090+In-T6. The results show a strong deformation texture for each alloy and indicate that both alloys were primarily unrecrystallized. A strong brass rolling texture component was observed at a plate thickness of T/2 and a cube texture, typical of recrystallization, was not present. These results indicate that partial recrystallization did not occur and is not a cause for the In effect on work hardening, but not yield strength.

TEM analysis indicates that the T_1 volume fraction was increased by In compared to 2090-T6. Messrs Kilmer and Mukhopadhyay conducted TEM analyses at UVa on

R2090+In-T6. Apart from confirming the subgrain structure of this alloy, this work established that the volume fraction of the T_1 strengthening phase was increased by indium compared to T6 2090. Presumably, the lack of an In effect on σ_{ys} is not explained by the gettering of In by constituent or dispersoid phases.

Optical and TEM microscopy established that a large amount of subboundary precipitates, and an associated δ'/T_1 precipitate free zone, formed in R2090+In-T6 after peak aging at 160°C. Subboundary precipitation was not significant in 2090-T8. TEM indicates that the boundary phase may be equilibrium T_2 ; the morphology was blocky, not plate-like, and EDAX evidenced the presence of Cu and Li in the particles. In was not detected in the EDAX spectra. These observations indicate that In was less effective than stretch deformation in promoting intra-subgranular precipitation, at least for aging at 160°C.

Experiments at LaRC demonstrated that subboundary precipitation formed during aging and not during possibly slow cooling from the solution treatment temperature. Specimens were solution treated, cold water quenched and aged for varying times at 160°C. Subboundary precipitates were not observed for the solution treated condition, but formed after 2 hours (or perhaps sooner) at 160°C, and increased in amount with increasing aging time. The microstructure which was produced by aging for 75 hours at 160°C was identical to that observed for the alloy which was solution treated by Reynolds Metals and similarly aged at LaRC. These results indicate that the subgrain boundary microstructure is a true characteristic of R2090+In-T6; the role of In and the aging response of the alloy at lower temperatures are unclear. Subgrain boundary precipitates have a strong adverse effect on fracture toughness.

Alloy Fracture Toughness: *J-integral fracture toughness testing was conducted at Fracture Technology Associates (FTA) and this capability was established at NASA-LaRC. Twelve specimens of R2090+In-T6, R2090-T8 and Vintage III Alcoa 2090-T8E41 (A2090-T81) plate were tested at FTA. Plane stress and plane strain fracture toughness experiments were conducted with L-T oriented compact tension specimens of varying thickness. Crack growth resistance was defined by an unloading compliance method according to standard methods. The "plane strain" specimens were sufficiently thick (11.9 mm) to satisfy the linear*

elastic criterion for valid K_{IC} and in some cases included sidegrooves. The "plane stress" specimens were sufficiently thick (1.5 mm) to satisfy the J-integral criterion for a plane strain initiation toughness, but were sufficiently thin to promote stable crack extension. The results from this testing are summarized in Table 1 and are discussed in an ensuing section.

The capability for conducting ambient and cryogenic J-integral fracture toughness experiments, based on an automated unloading compliance approach, has been established at NASA-LaRC. Duplicate plane strain specimens were tested at ambient temperature to compare with the results of the testing conducted at FTA. There was excellent agreement between the two laboratories. Testing at LaRC has also been conducted on specimens in the TL orientation in order to verify that adequate toughness properties were maintained in this orientation. As expected, the fracture toughnesses of A2090-T81 and R2090+In-T6 were lower in the TL orientation; this slight decrease is typical of Al-Li alloys. Cryogenic testing will commence during the next reporting period.

A2090-T81 exhibited the highest fracture toughness in both the plane stress and plane strain conditions. The reasonable crack initiation and growth toughnesses of A2090-T81 (Table 1) at ambient temperature are associated with a significant amount of delamination and transgranular shear (TGS) fracture. For the plane strain case, Figure 2 shows a metallographic cross section of the fractured specimen, perpendicular to the direction of crack growth. The sharply faceted crack surface is typical of cracking along $\{111\}$ slip planes in textured peak aged 2090. Delaminations, along high angle boundaries, are perpendicular to the main crack plane and are parallel to the crack growth direction. The level of delamination was significantly lower in the plane stress regime, however, fracture remained typically TGS in nature. The results for A2090-T81 in Figure 2 and Table 1 are consistent with the known roles of δ' localized slip and high angle boundary delamination^[4].

The fracture behavior of R2090+In-T6 is significantly degraded by subgrain boundary precipitates. Toughness is low and is characterized by intersubgranular (ISG) fracture without TGS or delamination. The data in Table 1 indicate that the fracture toughness of 2090+In-T6 is lower than that of A2090 for both specimen thicknesses. Fracture is characterized by ISG cracking, with a minimum amount of delamination, in both plane stress and plain strain conditions, Figure 3. The difference in toughness between A2090-T81 and

R2090+In-T6 is somewhat reduced in the plane stress regime due to the lack of extrinsic delamination toughening of A2090-T81 in plane stress.

ISG fracture in R2090+In-T6 is traceable to the high density of subboundary precipitates which form in the In bearing alloy, but not in 2090-T8. The different etching responses of the microstructures in Figures 2 and 3 indicate this difference in unrecrystallized subgrain boundary precipitation. ISG fracture is a low energy fracture event which presumably occurs prior to the onset of slip band cracking. The ISG crack path may be caused by either the boundary precipitates or the adjacent δ'/T_1 strengthening precipitate free zone.

In an effort to increase the toughness of R2090+In-T6, the alloy was resolution heat treated at 555°C for 0.5 hours, quenched in agitated ice water and aged at 160°C for 75 hours. Plane strain toughness testing of this material revealed no increase in toughness associated with the faster quench rate, compared to the vendor solution treated condition represented in Table 1. The primary fracture mode remained ISG. This result is consistent with optical microscopy observations on the occurrence of boundary precipitation during aging, but not after solution treatment. The question which remains to be answered is whether the high volume fraction of subboundary precipitates in 2090+In-T6 is due to prolonged aging (75 hours) at 160°C or is inherent with In additions to Al-Cu-Li alloys.

Future Research

During the next six months research will focus on the following areas:

- oo Analyze the crack growth resistance curves for all specimens tested at FTA, UVa and LaRC to define crack initiation and tearing modules toughnesses, with emphasis on the effect of stress state for the TGS/delamination and IGS fracture modes. These data will be compared to current fracture mechanics concepts which relate elastic and elastic-plastic characterizations and which have only been tested for ductile ferritic steels.
- oo Determine the fracture toughness and fracture mode of A2090-T81 and R2090+In-T6

at cryogenic temperatures for both the plane stress and plane strain cases. Examine the extent of delamination as a function of temperature and how it effects toughness. Define the proportions of TGS and IGS fracture.

- oo Investigate aging heat treatments to increase the toughness of R2090 + In in the T6 condition. Subboundary precipitate number density and morphology will be qualitatively determined as a function of aging time at 160°C and perhaps lower temperatures. Identify subgrain boundary precipitates and the associated PFZ in 2090+In-T6 compared to 2090-T6 and 2090-T8, and determine if the low toughness of 2090+In-T6 is a result of overaging in the absence of stretch or inherit with In additions.
- oo Examine mechanisms for the observation that the In addition to Al-Cu-Li-X alloys increases ultimate tensile strength, but has no effect on yield strength. Employ TEM to qualitatively assess T_1 number density and distribution in A2090-T81 and R2090+In-T6. Determine the work hardening behavior of these alloys.

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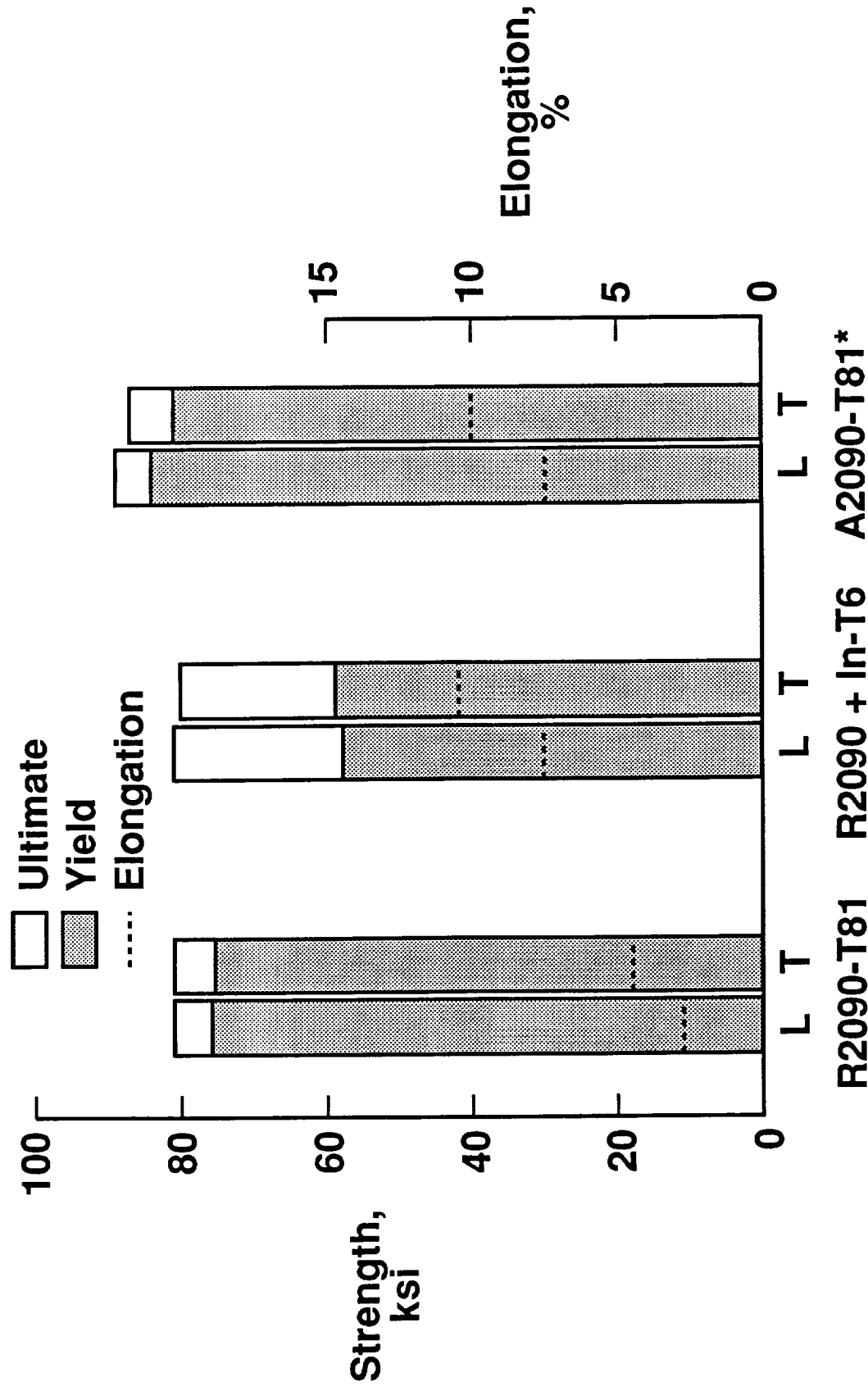
Table 1. Fracture mode and average J_{Ic} for plate 2090 at ambient temperature.

Material	Thickness (in.)	J_{Ic} (in.-lb/in. ²)	K_{Ic} (ksi $\sqrt{\text{in.}}$)	Plastic zone thickness	Amount of delamination	Primary fracture mode
A2090-T81	0.06	75	31	0.37	Medium	TGS/min. ISG
	0.47	56	27	0.03	High	TGS/min. ISG
R2090-T8	0.06	44	24	0.25	Low	ISG
	0.47	30*	20	0.02	Medium	ISG
R2090 + In-T6	0.06	55	27	0.57	Low	ISG
	0.47	32	21	0.05	Low	ISG

TGS \equiv transgranular shear

ISG \equiv intersubgranular

* Invalid according to ASTM E813



* Values from NIST

Figure 1. Tensile properties Al-Li-Cu-Zr alloys.



Figure 2. Cross section of A2090-T81 J-integral fracture toughness specimen.

Future plans

We propose to collaborate with Blankenship and Starke by characterizing the temperature dependence of the fracture toughness for each of these microstructures. Plate material has been purchased from Reynolds Metals for this purpose. Blankenship and Starke will determine the aging conditions that produce the two microstructures with similar T_1 size, morphology, and intragranular and subgrain boundary precipitate distributions. These workers will employ TEM to characterize the microstructures and deformation modes. Fatigue precracked compact tension specimens will be prepared in the L-T orientation for each microstructure; thickness will be 4 mm and width will be 50.8 mm.

Experiments at NASA-LaRC will measure the J-crack growth response, based on the automated unloading compliance method discussed in Program #3, for cryogenic (-196°C) to ambient temperatures. Experiments will be conducted at UVa to measure fracture toughness from ambient to elevated (250°C) temperatures with the electrical potential method discussed in Program #2. Fracture surfaces will be examined by scanning electron microscopy and metallographic cross sections.

The uniaxial tensile deformation behavior of WeldaliteTM 049 will be characterized at selected temperatures spanning the range from -196 to 300°C . The stress-strain relationship based on the Ramberg-Osgood power-law relation will be used to identify work hardening characteristics for uniform deformation prior to necking.

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Program 5 Measurements and Mechanisms of Localized Aqueous Corrosion in Aluminum-Lithium-Copper Alloys

Rudolph G. Buchheit, Jr. and Glenn E. Stoner

Objectives

The objective of this research is to characterize the localized corrosion and stress corrosion crack initiation behavior of Al-Li-Cu alloy 2090 in aqueous environments, and to gain an understanding of the role of local corrosion and occluded cell environments in the mechanisms of pitting and stress corrosion crack initiation and early-stage propagation.

Results

This research effort concluded in August, 1990 with the Ph.D. dissertation defense. The research objective, approach and progress are fully reported in two previous reports^[1,2] and in the dissertation^[3]. Major conclusions are reported below. This project will be continued as Task 6, "Mechanisms of Localized Corrosion in Al-Li-Cu Alloy 2090", as outlined in the latest renewal proposal^[4].

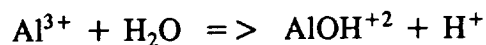
Conclusions

1. Three phases play a major role in localized corrosion and stress corrosion cracking of 2090 in aqueous environments. They are the T_1 phase precipitated on subgrain boundaries, Al-Cu-Fe impurity phases distributed throughout the plate, and the matrix phase. In order of decreasing nobility, these phases can be arranged as follows:

$$\text{Al-Cu-Fe} > \text{matrix-Al} > T_1.$$

Two types of pitting commonly observed during after exposure to aqueous chloride can be understood in terms of this arrangement.

- a.) Gross "constituent" particle pitting initiates by local galvanic attack of the matrix adjacent to the more noble particle.
 - b.) Preferential subgrain boundary corrosion occurs by selective dissolution of highly reactive T_1 concentrated on the boundaries.
2. Rapid stress corrosion cracking will occur when the potential applied to a specimen lies above the breakaway potential of the T_1 phase, but below that of the matrix phase. This is not satisfied in aerated 3.5 w/o NaCl solution, hence few SCC failures are observed under constant immersion conditions. The rapid SCC criterion can be satisfied in an aqueous environment containing Cl^- and a passivating anion like chromate or carbonate. Physically, the rapid SCC criterion implies that the crack walls comprised of matrix material are passive while the T_1 -rich crack tip can rapidly dissolve.
 3. Pits and crevices which form on 2090, immersed in an aerated bulk solution, are acidified with a pH of about 3.5 to 4. This is due to the following equilibrium hydrolysis reaction:



provided reduction reactions can occur freely on the external surface. In isolated crevices dissolution of lithium combined with consumption of H^+ by reduction inside the crevice causes alkaline crevice solutions to develop. Hydrolysis of lithium alone does not cause the crevice to become alkaline; rather it mitigates a pH increase in extremely alkaline crevice solutions.

4. Alloy 2090 is susceptible to pre-exposure embrittlement. During immersion in aerated NaCl solution, pitting and crevicing occurs. When the specimen is removed from the bulk solution, but still stressed, these pits retain electrolyte which becomes alkaline.

In the presence of atmospheric CO_2 , a passivating film forms on the walls of crevices creating a situation favorable for rapid stress corrosion cracking. The passivating film is a hydrotalcite-type compound whose stoichiometry is $\text{Li}_2(\text{Al}_2(\text{OH})_6)_2\cdot\text{CO}_3\cdot n\text{H}_2\text{O}$.

5. Pure aluminum can be passivated by immersion in one of several alkaline lithium salt solutions. This phenomenon appears to be related to a film formation process similar to that observed in alkaline Li_2CO_3 solutions.

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Program 6 The Effects of Zinc Additions on the Environmental Stability of Alloy 8090 (Al-Li-Cu-Mg-Zr)

Raymond J. Kilmer and G.E. Stoner

Objectives

The objectives for this PhD research are to document and characterize the effects that Zn additions have on the microstructure of alloy 8090 under different aging conditions and to correlate SCC behavioral changes with changes in alloy composition and microstructure. As an extension of this goal, emphasis will be placed on optimizing SCC behavior and alloy density.

Approach and Results

The program approach has been outlined in previous reports along with pertinent background information on the 8090 alloy system^[1,2]. The initial focus of this project has been directed towards documentation and characterization of four alloys whose nominal compositions fell within the 8090 composition window and had varying amounts of Zn additions up to 1.07 wt%. These alloys were specialty cast by Alcoa, rolled to 2.5 mm sheet and stretched to obtain a T3 condition. These sheet alloys were delivered in early April, 1990. Although no mechanical SCC tests were possible with these sheet alloys, it was felt that evaluation of the precipitation events both within the interiors of the grains and along the boundaries would add valuable insight as to the influence Zn additions have on alloy microstructure and that this information would be applicable to the understanding of the microstructural behavior of the plate alloys delivered in November of 1990 and January of 1991. Results of this work were reported in the literature^[3]. A brief summary of the pertinent findings are as follows:

- ** TEM revealed that Zn additions influence the subgrain boundary precipitation events in the alloys.

- ** Zn was found (via DSC) to partition to the δ' (Al_3Li), altering the volume fraction precipitating along with altering the enthalpic character of the δ' .
- ** The width of the δ' -free zone adjacent to the boundaries at peak aged condition was influenced by Zn content.
- ** Zn additions altered the morphology of the S' (Al_2CuMg) precipitates.

Stress corrosion cracking occurs intersubgranularly in alloy 8090 and subgrain boundary microstructure plays a central role in the phenomena. This early work already demonstrates one clear distinction between the alloys and likely plays a role in their relative SCC behaviors.

Current and Future Work

The four 44.5 mm 8090 + Zn variant plates are currently under evaluation. Aging curves for 160°C and 190°C have been obtained from these alloys and TEM is currently being undertaken to characterize their microstructures. As a means of initially screening the alloys to determine the extent of SCC susceptibility, a number of tests will be performed. Both alternate immersion and breaking load experiments will be performed on S-T cylindrical tensile samples. Two aging conditions will be evaluated; an underage temper (5 hours at 160°C) and a peak age temper (100 hours at 160°C). The alternate immersion tests, following the ASTM G-44 standard will be employed as a pass/fail test to determine the optimum conditions for breaking load experiments. Currently two different exposure stresses are planned, their values dependent on the results of the AI tests. S-L and S-T toughness and tensile tests on the four alloys for the two aging conditions will be performed prior to the experiments to aid in setup and analysis of the experiments. It is felt that these techniques will provide the most useful information on differences in SCC behavior among the compositions. The breaking load technique provides a numerical measure of SCC damage and allows for a more quantitative fracture mechanics-type approach. The plates have been sectioned and are now being aged and a trip to Alcoa in late January is planned. These tests

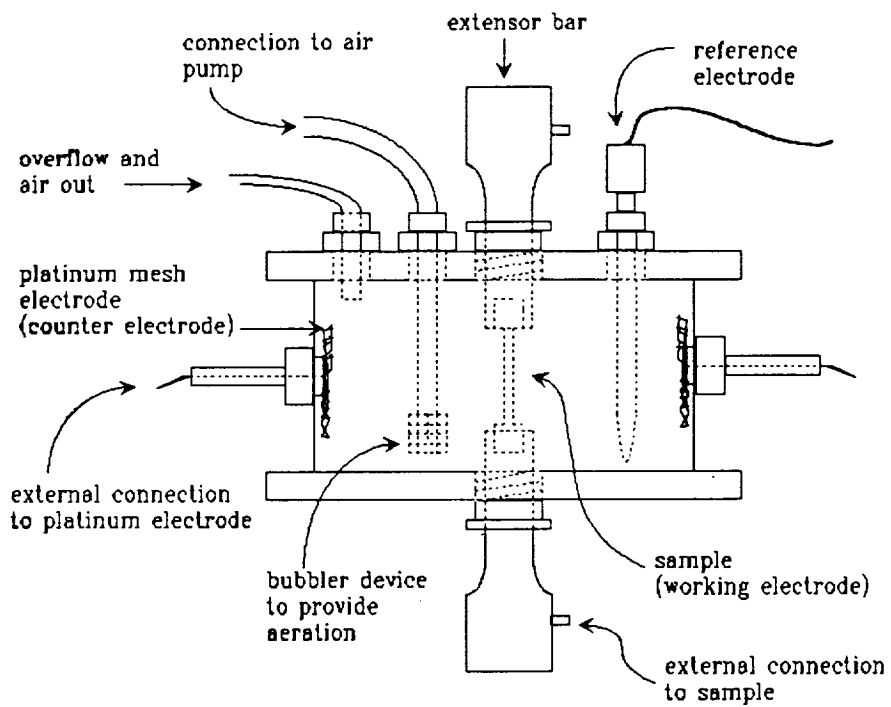


Figure 1. Schematic of load cell for time-to-failure experiments

Program 7 Deformation and Fracture of Aluminum-Lithium Alloys: The Effect of Dissolved Hydrogen

F.C. Rivet and R.E. Swanson

Department of Materials Engineering
Virginia Polytechnic Institute and State University

Objective

The objective of this study is to characterize and understand the effects of hydrogen on the deformation and fracture behavior of 2090 and 2219, especially at low temperatures. Additionally, 8090 and Weldalite will be included in this program.

Results to Date

Mr. Rivet has completed his research and submitted a thesis to Virginia Polytechnic Institute in partial fulfillment of the Master of Science Degree in Materials Engineering and under the supervision of Professor Robert E. Swanson. The following material is reproduced verbatim from this thesis.

Abstract

The objective of this work is to study the effects of dissolved hydrogen on the mechanical properties of aluminum-lithium alloys: 2090, 2091 and Weldalite™ 049, and to compare with the effects on aluminum-copper 2219 alloy. Prior to mechanical testing, aging studies were performed for 2090 and 2219 using microhardness Vickers to determine the peak aged conditions required by NASA. The Charpy tests are part of this study designed to investigate the effects of temperature and notch orientation on fracture behavior. Disk rupture tests were used with various gases (hydrogen and nitrogen) and three strain rates (increament [sic] of 50 psi every 20, 200 and 300 seconds) and two temperatures (room and liquid nitrogen temperatures) to determine the effects of hydrogen on the sample during the

tests. Some independent studies on the corrosion behavior and electrochemical hydrogen charging of 2219 and 2090 were also performed.

An effect of double peak aged condition was found for both 2219 and 2090 alloys. Prior to mechanical testing, the 2090 received in the T3 or W51 conditions was chosen to be aged in an air furnace at 170°C for 16 hours. The Charpy studies showed a higher propagation energy needed for the T-S and L-S orientations than for the L-T and T-L orientations, due in large part to the extensive delamination propagation of the fracture. The disk rupture tests showed an important decrease of the fracture (strain - [sic]) to failure on the 2090 and 2091 due to hydrogen while no important variations were seen for the 2219 and the Weldalite™ 049 alloys. No effect of hydrogen were [sic] found, with the disk rupture test, at cryogenic temperature and for all alloys. The corrosion behavior of 2219, as well as 2090, showed development of pits under neutral and acidic environments while general corrosion was obtained [sic] with basic environment. Two solutions were found to charge the samples in hydrogen: a potentiostatic test for 5 hours at -3 V, and a galvanostatic test for 20 hours at -500 μ A, both performed in a 0.04 N HCl plus As₂O₃ environment.

Conclusion

The aging sequence of both 2219 and 2090 show a double peak aged condition explained by different precipitation occurring in the alloys. The time required to bring up the 2090 T3 and W51 alloys to the peak aged condition corresponds to 16 hours at a temperature of 170°C. These two alloys also gave the double peak condition.

The disk rupture tests have shown a dramatic effect due to hydrogen during the experiments for both 2090 and 2219 at room temperature, while no difference appeared at cryogenic temperature. For these two alloys, hydrogen was increasing the amount of transgranular fracture from nearly 0% under nitrogen testing to about 20% under hydrogen. No or nearly no effects of hydrogen was [sic] noticed for Weldalite™ 049 and 2219 alloys.

The possibilities of replacing 2219 by a [sic] Al-Li alloy does not give any doubt for the Weldalite™ 049 [sic], while more studies will be needed to simulate the real conditions of a hydrogen tank to evaluate 2090 and 2091.

Program 8 Investigation of the Reaction Kinetics Between SiC Fibers and Selectively Alloyed Titanium Matrix Composites and Determination of Their Mechanical Properties

Douglas B. Gundel and F.E. Wawner

Objective

The objective of this study is to define the reaction kinetics between SiC fibers with different surface morphologies and titanium matrices containing different structural phases, and to determine the effect of the reaction on the mechanical properties of the composites. Ti-1100 is the primary matrix of interest in this study.

Approach

The approach to be taken in this study is to fabricate titanium matrix composites using titanium that has been selectively alloyed to generate improved high temperature properties, and silicon carbide fibers as the reinforcement. The matrix alloy of immediate interest is the recently developed, near-alpha (α) alloy designated Ti-1100 (Ti-6Al-2.8Sn-4Zr-0.4Mo-0.45Si). This alloy is a derivative of Ti-6242-Si, but is reported to have better creep properties, thermal stability and ductility. Of equal interest is the new metastable beta (β) alloy BETA 21S (Ti-15Mo-2.7Nb-3Al-0.2Si) which has similar properties to Ti-15-3, but is reported to have improved oxidation resistance. For comparison of reaction kinetics, samples are also being produced using Ti-15V-3Cr-3Al-3Sn (a β alloy), Ti-6Al-4V (an $\alpha + \beta$ alloy), Ti-14Al-21Nb (an $\alpha-2 + \beta$ alloy), and commercially pure titanium.

SCS-type SiC fibers manufactured by Textron Specialty Materials (Lowell, MA) with the designations SCS-0 (uncoated), SCS-6 and SCS-9 are the reinforcements used in the experiments. Selective coatings are also being applied to these fibers to evaluate their effectiveness as diffusion barriers.

Progress During Reporting Period

Work during the past reporting period concentrated on evaluation of the kinetics SCS-

6 fibers in a BETA 21S matrix, preparation of a Master of Science Thesis^[1], and a technical progress report^[2] summarizing all of the work to date on the kinetics of the Ti alloy/SiC reactions. Conclusions from this work are presented below.

The reaction kinetics of UA Ti, Ti-6-4, Ti-15-3, BETA 21S, Ti-1100, and Ti-24-11 with SCS-6 fibers were determined in the temperature range of 800 to 1000°C. Ti-1100 was found to be the slowest reacting conventional matrix, while Ti-24-11 was the slowest reacting overall. Alloy additions were found to reduce the reaction kinetics in all of the cases studied. The experimentally determined reaction kinetics were found to predict the size of the reaction zone after exposure for 1000 hours at 700°C for the Ti-1100, Ti-24-11, Ti-6-4, and UA Ti systems, but not for Ti-15-3.

The α (hcp) and β (bcc) phases of titanium were found to have slightly different kinetic parameters in reaction with the fibers. Extrapolation of the data suggests that above 910°C the α phase will react faster than the β phase, with the reverse true below this temperature. Only in an alloy will both phases coexist, however, in which case the compositional difference between the phases may also influence their reactivities.

Various other fibers were also evaluated in these matrices. Due to the presence of a similar coating on its surface, the SCS-9 fiber was found to react at the same rate as SCS-6 until its coating had been consumed. At this point the reaction rate sometimes changed, as did the composition and morphology of the reaction products. The TiB₂/SCS-6 fibers were found to react at nearly the same rate as the SCS-6 fiber. The 1 μ m TiB₂ coating did not seem to have any significant effect on the reaction rates. Silicon carbide fibers that did not have an SCS coating on the surface (SCS-0) exhibited slightly different reaction kinetics than SCS-6 depending on the matrix.

A β -depleted zone formed and grew in the matrix adjacent to the fiber in the Ti-24-11/(SCS-6, SCS-9, SCS-0, TiB₂/SCS-6) systems. This zone was often observed to contain cracks extending from the fiber-matrix interface to the edge of the β -depleted zone. The size of this zone was successfully reduced or eliminated by coating the fiber with vanadium (1 μ m) or tungsten (10 μ m) prior to composite fabrication. Presumably, the reason for this is the β -stabilizing nature of these elements.

Several observations in this research led to the formulation of a possible mechanism

for the growth of the reaction zone in Ti/SiC systems. Growth may be controlled by the transport of titanium atoms through the reaction zone to the fiber-reaction product interface. A simple mathematical model was developed that gave the proper (parabolic) dependence of the reaction zone size on time.

Using this model, the effective diffusivities of titanium in the reaction zone were calculated at various temperatures. The effective diffusivities were much faster than tracer diffusivities of titanium in titanium carbide extrapolated from higher temperatures.

Future work will emphasize kinetic measurements utilizing the Sigma SiC fiber (TiB_2 coated), and extended mechanical property measurements of Ti-1100 and BETA 21S/SiC composites as has been described in the renewal proposal for Grant No. NAG-1-745.

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Program 9 Quantitative Characterization of the Spatial Distribution of Particles in Materials: Application to Materials Processing

J.B. Parse and J.A. Wert

Introduction

The spatial distribution of second phase particles is of interest in two broad areas of materials science: understanding the relationship between microstructure and bulk properties (toughness, ductility, strength), and tailoring a process to produce a particular microstructure. In either case, a method for characterizing the distribution of second phase particles is a key component of the overall problem. To date, materials scientists have used predominately qualitative methods to describe the distribution of second phase particles in materials ("uniform", "clustered" or "banded", for example). These qualitative descriptions are unsatisfactory for incorporation into models of material behavior.

Some previous investigations have sought to use quantitative methods to describe the spatial distribution of particles in materials^[1,2]. In all cases, the resulting information has been applied to the problem of understanding the relationship between microstructure and material properties. We are unaware of any prior systematic investigation into the relationship between processing parameters and the spatial distribution of second phase particles.

Objective

The objective of the present investigation is development of a broadly-applicable method for the quantitative analysis and description of the spatial distribution of second phase particles. This method should characterize the spatial distribution and its inhomogeneities in a manner which is of use to the materials scientist. As the method is intended to be available to a wide range of researchers in materials science, it has been designed to operate on a desktop computer. A second objective is application of the method for characterizing second phase particle distributions in a materials processing problem. The problem we have selected in consultation with the NASA technical monitor is understanding the effect of consolidation

processing parameters on oxide particle distributions in a PM aluminum alloy.

Method for Analyzing Second Phase Particle Distributions

After consideration of several techniques, the Dirichlet tessellation technique was selected as the basis for the analysis method. A brief description of this technique follows; more details may be found in previous reports^[3,4].

The Dirichlet tessellation for an array of N points in two dimensions is the sum of the Voronoi polygons for each of the N points in the array. The Voronoi polygon for each point is constructed in the following manner:

- i) draw a line from the chosen point to every other point in the array
- ii) erect the perpendicular bisector to each of these lines
- iii) the Voronoi polygon is the smallest convex polygon formed by the intersection of the perpendicular bisectors.

Each Voronoi polygon circumscribes that area which is closer to its generating center (central point) than to any other point in the array. The set of Voronoi polygons comprising the Dirichlet tessellation of a point array is space-filling and unique. Geometrical considerations require certain mathematical relations between some polygon and tessellation properties. The Dirichlet (or primary) tessellation is an area-based representation of the point array; a mathematically equivalent, distance-based representation is known as the dual tessellation. Several polygon properties are of interest to the materials scientist; these include polygon area (inversely related to local area fraction) and near-neighbor distances.

The Voronoi polygon described above is generated by a point in the mathematical sense. The second phase particles whose spatial distribution is our objective have finite size and variable shape. Adaptations of the Voronoi polygon technique from zero-dimensional points to finite particles of varying morphology have been developed^[2]; such approaches are extremely computation-intensive and are typically implemented on a supercomputer. Since one of the objectives of the present research project is to develop a widely applicable technique and to limit computing requirements, no attempt has been made to include particle

size or morphology in current versions of the analysis method.

Second phase particle position data from a micrograph may be input into the analysis system through a digitizing tablet. Information generated by an image analysis system would also be suitable if put in the appropriate format (sequential file). During development of the analysis programs, a wide variety of computer-generated point distributions exhibiting well-characterized types and degrees of inhomogeneities were used to test the analysis programs and evaluate their performance.

Several computer programs were developed to carry out the various phases of the analysis, as memory limitations precluded incorporation of all parts of the analysis into one master program. The Dirichlet tessellation of a point array, which is the basis for all further phases of the analysis, is generated by a dedicated program. This is a computation-intensive process, with the time required going as N^2 , for N particles. This process need be performed only once, as the program outputs a data file describing the Voronoi polygons and related tessellations.

The tessellation data file mentioned above serves as the input for the various analysis programs. One program analyzes various properties of the Voronoi polygons representing individual particles in the point array. These properties, including polygon area, aspect ratio, orientation and others, characterize to a great extent the local environment of each particle. A second program analyses properties of the Delauney triangles comprising the dual tessellation, which effectively represent the vector distances between near-neighbor particles. Properties of interest derived from this representation include distances to: nearest neighbor, furthest near-neighbor, mean neighbor distance and others. These properties characterize the local environments of individual particles in distance-based terms, distinct from the area-based parameters derived from the primary tessellation. These two representations are mathematically equivalent^[3]; either may be generated from the other. The object of using both is that each emphasizes a different aspect (area or distance) of the particle distribution, and is thus more or less applicable to varying problems.

The data derived in these programs may be displayed in several plotting formats, including cumulative fraction or cumulative probability versus linear or logarithmic scaling of the property selected. The on-screen plot display includes two vertical-bar cursors which

allow selection of those particles whose properties fall between the two cursors; the polygons thus selected may be color-highlighted in a graphical display of the tessellation.

The analysis programs described above evaluate only properties of individual second phase particles; grouping of particles into inhomogeneities such as stringers or clusters is not considered. As inhomogeneities of this type are of interest to the materials scientist, a program was developed which characterizes the degree of clustering in a particle distribution and evaluates distribution properties such as number and size of clusters, approximate aspect ratio and orientation of clusters, closest vector distance to near clusters, and others. To characterize clustering in a particle array, one must first develop a working definition of a 'cluster'. This program requires the user to specify a maximum separation for the association of a point with a cluster (the 'cluster-cutoff distance') and a minimum number of points (2,3,4...) necessary for consideration of a group of points as a cluster. The analytical method used by the program is based on the dual tessellation representation of the data; the program uses the same input file and has the same data display options as those described above. A plot of a property such as mean near-neighbor distance is convenient for selection of the cluster cutoff distance, which is specified by positioning a cursor on the graph at the desired value.

As mentioned above, the performance of the analysis programs was validated using computer-generated particle distributions. Preliminary application of these techniques to particle distributions found in real materials (Al-SiC composites, RS Al-Fe-Si-V and Al-Mn-Si PM alloys) demonstrated that the method provided a satisfactory analysis for materials of these types. It was determined that a systematic study of the spatial distribution of second-phase particles in a particular material should be performed to demonstrate the analysis technique. Previous work in this area has focussed on the interaction between the spatial distribution of particles and material properties. We have elected to focus the present investigation on the relationship between processing parameters and the second phase particle distributions, a relatively unexplored topic.

Progress During the Current Reporting Period

After consideration of several candidate materials and processes for a systematic

investigation of the effects of processing parameters on second phase particle distributions, a rapidly solidified Al alloy (Al-2Mn-1.2Si-0.4V-0.1Zr) under investigation at NASA Langley was selected. This alloy contains Mn-silicides and is a candidate material for high temperature applications. Oxide stringers in PM-Al materials can degrade mechanical properties such as toughness and are a suitable subject for this analysis method. The investigation will consist of producing sheet material via a PM process, using a variety of processing conditions and then analyzing the spatial distribution of oxide particles in the material produced. The aluminum alloy powders are to be produced at NASA Langley and the general sequence of processing steps will be similar to that used at NASA Langley:

- i) cold compact to approximately 70% density
- ii) vacuum hot press to full density
- iii) upset forge (hot)
- iv) hot roll to sheet (approximately 95% total reduction)

Processing equipment available at UVA is capable of duplicating the range of process conditions used at NASA. This allows the production of small specimens (1" dia. x 1/2" compacts) at UVA, increasing the number of experimental conditions that may be analyzed from a limited quantity of powder. It is anticipated that two or three processing conditions will ultimately be selected for the production of larger samples (3" dia. x 1.5" compacts) at NASA Langley. Mechanical properties of this material (measured at NASA) will be correlated with the observed spatial distribution of oxide particles.

The processing conditions chosen as experimental variables were:

- i) Hot working temperature for forging and rolling (T) (3 values; 800, 850, 900°F)
- ii) Ratio of forging to rolling in total reduction (F/R) (3 values; F/R = 0.6, 1.0, 1.4)
- iii) Surface state of powders (as-received, intentionally-oxidized, intentionally-hydrated).

An estimated 21 sets of experimental conditions (7 combinations of T & F/R at each of three powder surface states) will be evaluated by the analysis method on small samples produced

at UVA.

A die assembly for the vacuum hot press at UVA was designed and fabricated from Inconel 625. A double-acting configuration was chosen for ease of compact ejection and more uniform density. A schematic view of the die assembly is shown in Figure 3. There is a significant clearance between the two rams and the cylinder wall to eliminate the possibility of seizing; pressure plates on the face of each ram provide a close tolerance to prevent back-extrusion of metal during hot pressing and are attached to the rams via a 'break-away' connection to facilitate disassembly of the apparatus in the event of adhesion between the compact and the die.

A small upset forging apparatus was also designed and fabricated. The apparatus consists of two Inconel platens approximately 2" square held in a stainless steel frame; the entire assembly, including compact and guides, is heated in a muffle furnace to the desired temperature. Once the desired temperature has been reached, the assembly is placed in a 200 ton hydraulic press and upset forged to the desired reduction.

For the purpose of process development and troubleshooting, a small quantity of a similar alloy powder (NASA designation 'A3') was obtained. Samples of this powder ($106\text{ }\mu\text{m}$ > powder particle diameter > $75\text{ }\mu\text{m}$) were cold compacted at approximately 5,000 psi to approximately 70% density in both the as-received and intentionally-oxidized (1 hr @ 600°F in air) conditions. The cold compacts had sufficient strength for handling; autopicnometer measurements indicated that all porosity present was open.

The compacts were vacuum hot pressed at 850°F for 30 min. in a vacuum of approximately 90 mtorr. Maximum hot pressing pressure was 15,000 psi, yielding approximately full density for the intentionally-oxidized sample. Graphite spray provided a satisfactory anti-seize effect. These specimens (1" dia. x 1/2") were then hot-upset at 800°F to 1.45" dia. x 1/4" under 15,000 lbf and rolled to 0.026" sheet, for the desired reduction in thickness of 95%. This represents the lowest hot working temperature envisioned in the experimental program; fortunately, minimal edge cracking was noted in both the as-received and the intentionally-oxidized material. Scanning electron micrographs of the central section of the sheet produced are shown in Figure 4. The oxide stringers shown are the largest observed in approximately a 6" length of section.

Conclusion and Future Work

During the current reporting period, an alloy-PM process system was chosen for a systematic study of the effects of processing parameters on the spatial distribution of second phase particles. The alloy selected (Al-2Mn-1.2Si-.4V-.1Zr) is a candidate for high temperature applications under investigation at NASA Langley. The distribution of oxide particles in stringers will be analyzed and correlated with processing parameters. An apparatus allowing duplication of the NASA processing sequence at UVA was designed and fabricated. The equipment was successfully tested with powder of a similar alloy composition using the most difficult processing condition envisioned. Preliminary electron microscopy revealed significantly larger stringers in samples fabricated from the intentionally-oxidized powder than in those produced from as-received powder. Sufficient powder for systematic investigation of the effect of consolidation processing parameters on oxide particle distributions has been produced at NASA Langley.

Tasks to be performed during the final year of this research project include: production of the small specimens using the desired process conditions; analysis of the spatial distribution of oxide particles in the material produced; selection, based on this analysis, of two or three sets of processing conditions for the production of large samples at NASA Langley; mechanical property testing at NASA (tensile and Kahn-tear toughness); and correlation of toughness with the spatial distribution of oxides resulting from different processing conditions.

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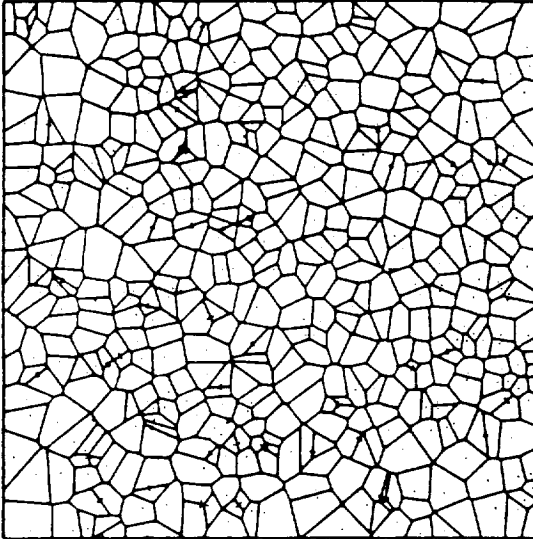
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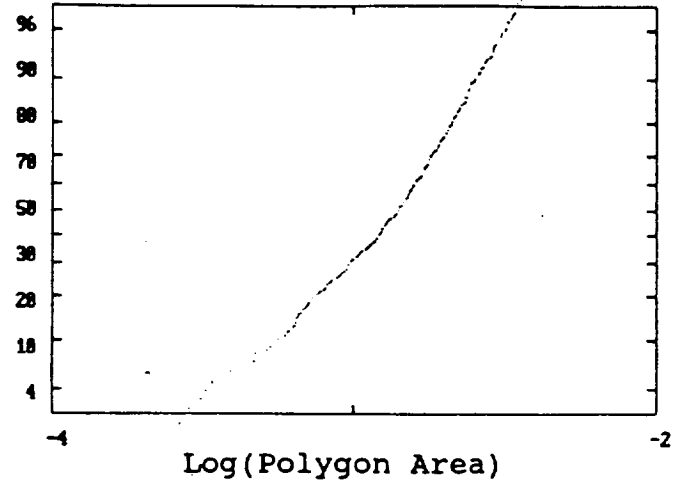
TESSELLATION-BASED ANALYSIS

Primary Tessellation

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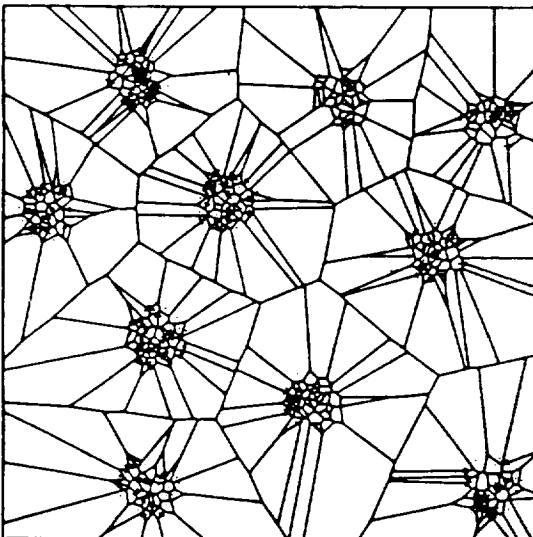


Cumulative
Probability

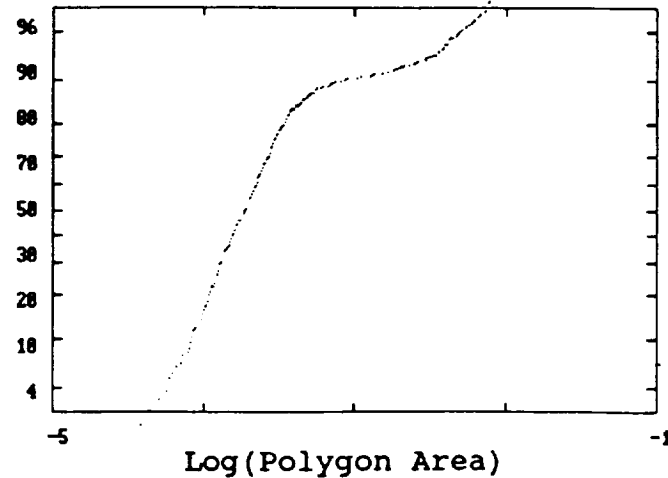


-----RANDOM ARRAY-----

Primary Tessellation: 31825



Cumulative
Probability

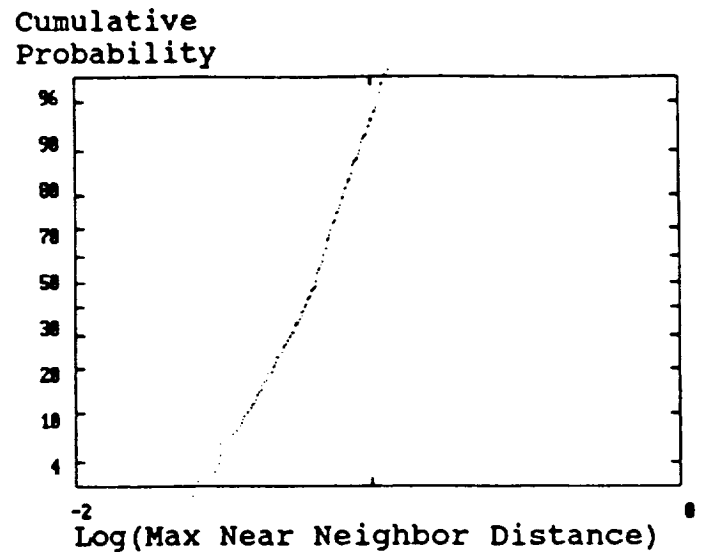
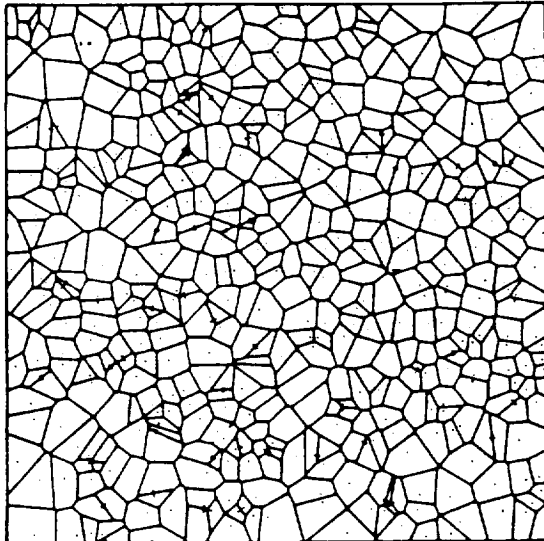


-----CLUSTERED ARRAY-----

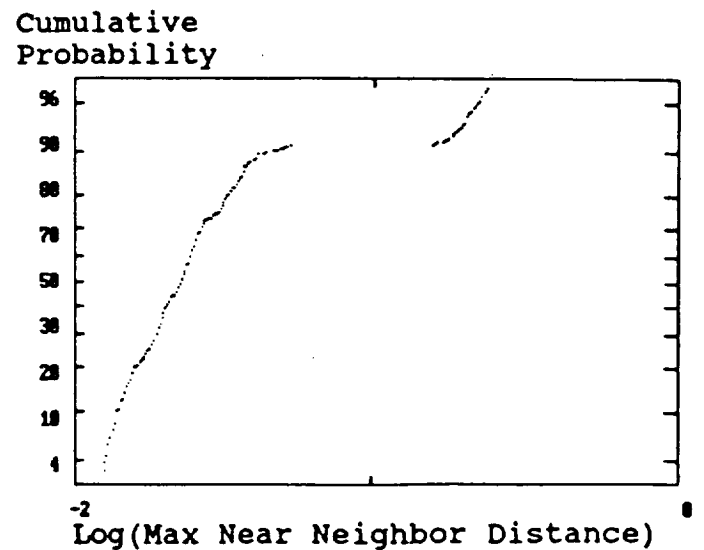
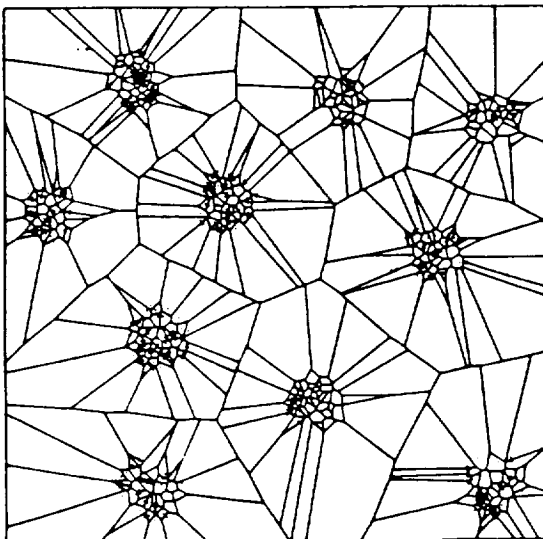
Figure 1.

TESSELLATION-BASED ANALYSIS

Dual Tessellation



-----RANDOM ARRAY-----



-----CLUSTERED ARRAY-----

Figure 2.

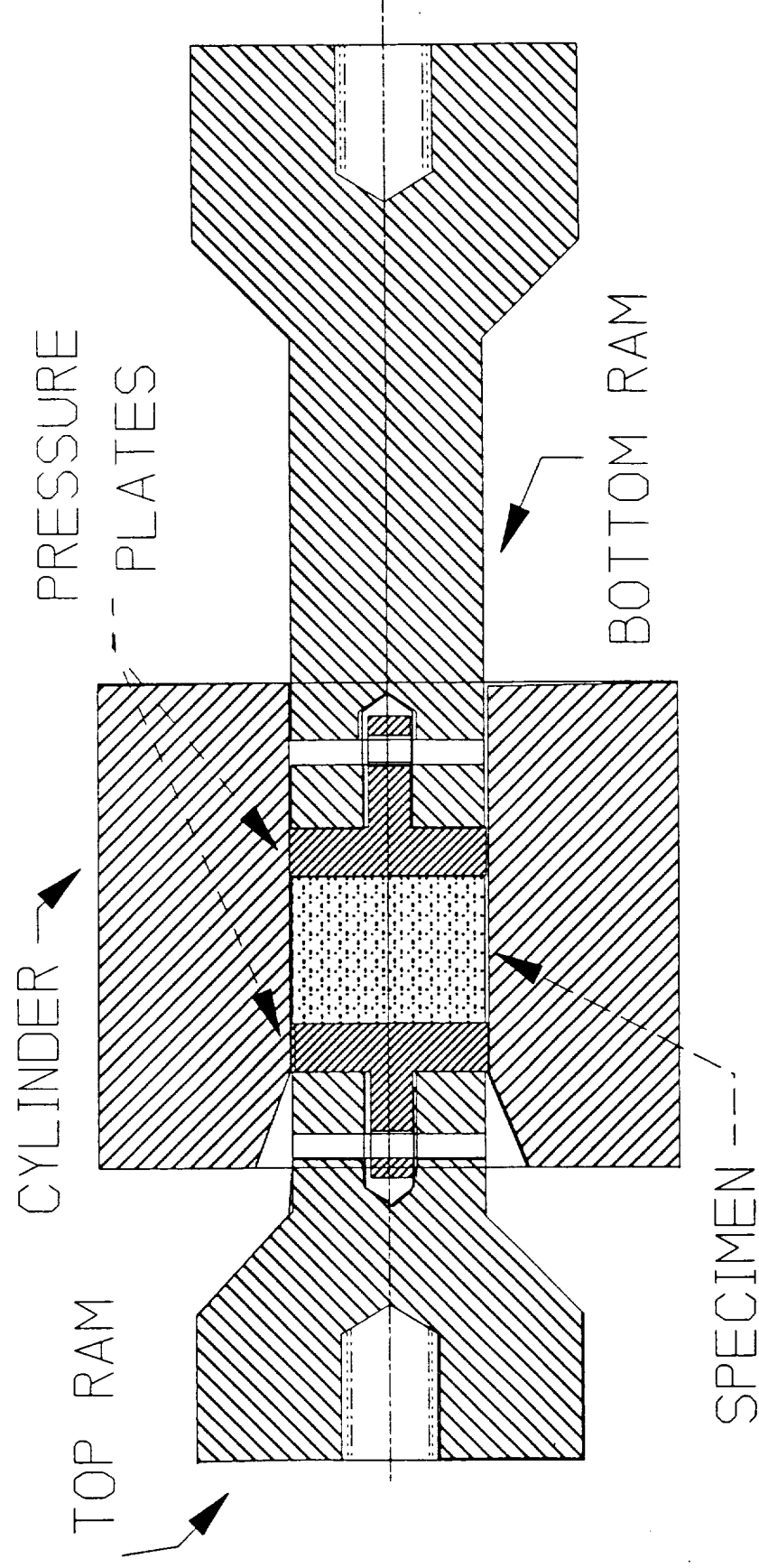


Figure 3. Hot Pressing Die Assembly

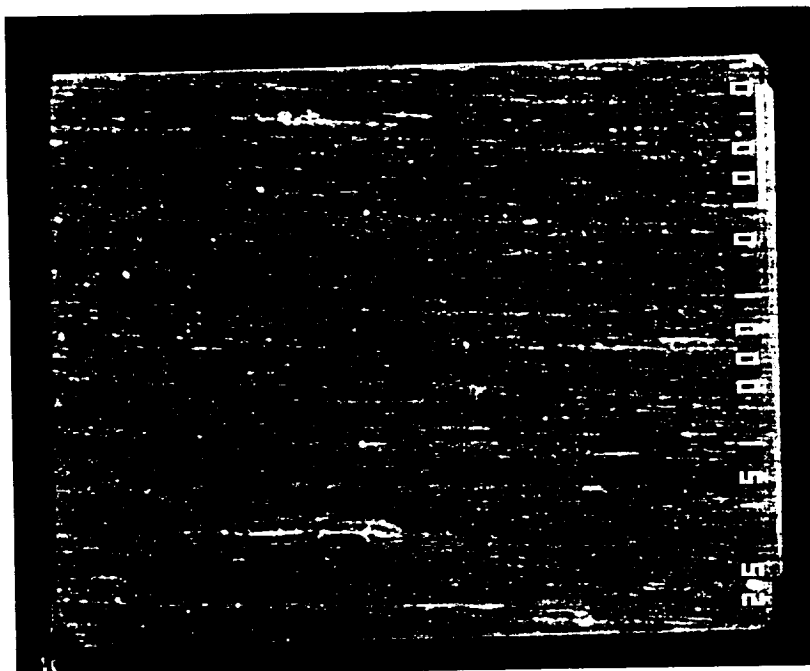


Fig. (4a): Sheet fabricated from as-received powders (150x).



Fig. (4b): Sheet fabricated from intentionally-oxidized powders (150x).

Program 9 **Inelastic Response of Metal Matrix Composites Under Biaxial Loading**

C.J. Lissenden, F. Mirzadeh, M.-J. Pindera and C.T. Herakovich

Objectives

The long-term objective of this investigation is aimed at attaining a complete understanding of the inelastic response of metal matrix composites subjected to arbitrary, biaxial load histories. The core of the research program is a series of biaxial tests conducted on different types of advanced metal matrix composite systems using the combined axial/torsional hydraulic load frame in the Composite Mechanics Laboratory at the University. These tests involve primarily tubular specimens and include tension, compression, torsion and combinations of the above load histories in order to critically assess the inelastic response of advanced metal matrix composites in a wide temperature range.

Approach

The approach employed to characterize the inelastic response of advanced MMC over a wide temperature range and arbitrary biaxial loading consists of combined experimental and analytical programs. The experimental program involves two stages. The first stage entails development of a test method most appropriate for the given MMC system selected. This includes the design of test fixtures and identification of strain measurement techniques followed by pilot tests and subsequent correlation of preliminary results with those obtained using other techniques. The actual test program is carried out in the second stage and depends on the particular aspect of the inelastic response to be addressed. For example, characterization of the elastoplastic response of a given MMC will first involve tests aimed at establishing the shape of the yield surface under biaxial loading, followed by tests aimed at establishing the actual plastic stress-strain law for arbitrary loading.

The analytical program involves development of models to predict initial yielding and subsequent inelastic response of MMC using a combined micromechanics approach and laminated plate and tube analysis^[1-9]. The micromechanics approach is based on the method

of cells model that has the capability of generating the effective response of metal matrix composites in the linear and inelastic range in the presence of temperature-dependent properties of the individual constituents and imperfect bonding. Micromechanical predictions for the response of a unidirectional ply subsequently will be employed as input in macroscopic laminate analyses to predict the response of multidirectional composite tubes under combined loading.

The analytical predictions will, in turn, be employed in identifying load histories aimed at critically testing the predictive capabilities of the developed models under the most general loading conditions. The last stage of the experimental/analytical sequence is an iterative one. It is repeated until satisfactory convergence between theory and experiment, and thus basic understanding of the mechanisms responsible for inelastic effects, is attained.

Synopsis of Results to Date

Mr. Clifford J. Lissenden, a PhD candidate in Civil Engineering/Applied Mechanics, started his work on the program in September, 1990. Ten SCS-6/Ti-15-3 tubes, six of which are angle-ply [$\pm 45^\circ$]_s configurations and four of which are [0°] configurations, have been secured from McDonnell Douglas for the test program. Test fixtures have been designed, fabricated and tested for 4" and 1.5" tubes. Data acquisition systems have been enhanced to acquire the necessary stress, strain and displacement data, and to display the data graphically in real time during the tests. Preliminary tests have been conducted to verify the test method and to measure the elastic properties of the composite tubes. These results are summarized in Table 1.

A major effort has been also devoted to the micromechanics predictions. Two micromechanics programs have been enhanced to provide predictions of initial yield surfaces for unidirectional and laminated MMC and subsequent nonlinear stress-strain response. Examples of the nonlinear stress-strain response and yield surfaces generated with the enhanced programs are given in Figure 1. Comparison of the preliminary experimental data given in Table 1 with the predictions of the micromechanics models is included in the table. The micromechanics programs are now being exercised to provide predictions for a variety of axial/torsional load ratios. The load ratios to be used in the testing program will be based

in part on these theoretical predictions. Those load ratios that provide the most critical test of the theories and the most unusual response will be included in the test program.

A meeting between the UVa researchers and Dr. Steve Johnson of NASA Langley and Mr. Dung Ngo of McDonnell Douglas was held at the University to review the status of the project to date and to develop a plan for the testing program. A number of possible loading paths has been discussed with the view of capturing the important response features of the given composite system. A method of testing for the effect of interfacial debonding observed in this material on the biaxial response was suggested. Tests are currently under way to investigate this, as well as other aspects of the combined response of SiC/Ti tubes.

Significance

The preliminary results generated thus far with the micromechanics model illustrate the effect of residual stresses and imperfect bonding on initial yield surfaces and inelastic response of $[0^\circ]$ and $[\pm 45^\circ]$, SCS-6/Ti-15-3 laminates loaded by different combinations of stresses. Validation and/or enhancement of the predictive capability of the employed micromechanics scheme will provide the analyst/designer with a powerful technique to study the effect of various micro-variables on the response of metal matrix composites under a variety of combined loading conditions.

Future Work

The 1991 effort will be devoted primarily to conducting tests on the tubes and correlating the experimental results with the predictions of the micromechanics model and laminate analysis. Laminate analysis work will include modification of an existing computer code that has been developed to analyze the nonlinear response of polymeric matrix composite tubes under arbitrary axisymmetric loading. The modification will entail incorporation of the micromechanics model for the individual layers of a laminated tube.

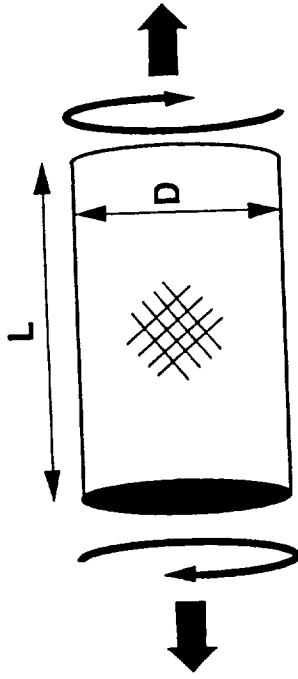
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- (1) Derstine, M. S. and Pindera, M-J., "Nonlinear Response of Composite Tubes Under Combined Thermomechanical Loading," in Composites and Other New Materials for PVP : Design and Analysis Considerations, ASME PVP-Vol 174 , D. Hui, T. J. Kozik, G. E. O. Widera and M. Shiratori, Eds., pp. 19-28, 1989.
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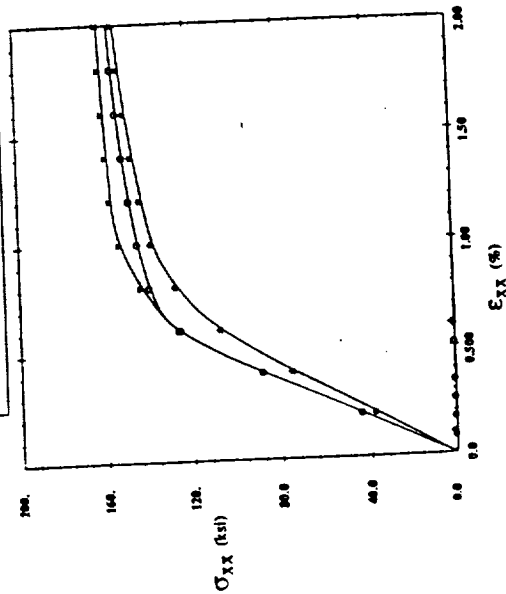
Table 1 Material Properties				
	[0] ₄		[±45] _s	
	Predicted	Measured	Predicted	Measured
$E_x(\text{MSI})$	31.2	NA	23.3	17.3
$G_{xy}(\text{MSI})$	8.13	NA	10.5	8.82
ν_{xy}	0.313	NA	0.375	0.377
$G_{12}(\text{MSI})$	8.13	NA	7.86	6.29

Inelastic Response of Metal Matrix Composites Under Biaxial Loading

Material: SCS-6/Ti 15-3
 Tube Layups: $[\pm 45]_s$ $D=4"$ $L=12"$
 $[0]_s$ $D=1.5"$ $L=7"$



SCS-6/Ti 15-3
 $(\pm 45)_s$, $V_f=0.4$
 $\Delta T=0^\circ\text{F}$, $R_c=0$, $R_t=0$
 $\Delta T=-1800^\circ\text{F}$, $R_c=0$, $R_t=0$
 $\Delta T=-1800^\circ\text{F}$, $R_c=6E-6$, $R_t=15E-6$



SCS-6/Ti 15-3
 $[0]_s$, $V_f=0.4$
 $\Delta T=0^\circ\text{F}$, $R_c=0$, $R_t=0$
 $\Delta T=-1800^\circ\text{F}$, $R_c=0$, $R_t=0$
 $\Delta T=-1800^\circ\text{F}$, $R_c=6E-6$, $R_t=15E-6$

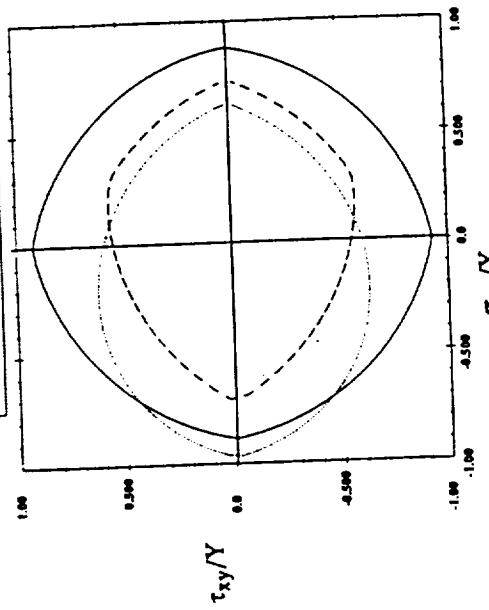


Figure 1

Program 11 **Design of Cryogenic Tanks for Space Vehicles**

Charles Copper, W.D. Pilkey and J.K. Haviland

Objectives

During the year 1990, the research objective was to investigate a new structural design for the tanks of large launch vehicles, using the Advanced Launch Vehicle (ALS) as an example. The proposed design consisted of superplastically formed corrugated hat-section stringers and frames in place of integrally machined stringers and separate heavy frames. The stringers and frames would be manufactured in subassemblies which would be spot welded to the skins.

The objective will change for the year 1991 to an investigation of thermal problems in shells such as those that could be used in the hydrogen tanks of the National Aerospace Plane (NASP) or in the fuselage sections of high speed civil transports. Such thermal problems would arise when a shell is subjected to external heating while the internal walls are maintained at the temperatures of cryogenic fluids. Depending on the particular design, the tank could be load carrying, subject to compression and bending loads. Insulation could compound the problem by increasing the thermal stresses, such as in single walled shells with internal or external insulation, or in double-walled shells separated by sandwich materials. Severe thermal stresses could also be induced by internal stiffeners and frames. We would work with the Materials Science Department in the selection of new candidate materials.

Method of Analysis

During the past year, the "Computational Analysis Testbed" (COMET) program has been used to analyze the buckling strength of the cryogenic tanks of the ALS. This program is available at NASA Langley Research Center (LaRC) on VAX and CONVEX computers in the Computational Mechanics Branch. To use this program, inputs are first processed on the PRIME computer at the Computer Aided Engineering (CAE) facility in the UVA Albert Small building, using the PATRAN program. These inputs are then transmitted by TELNET

to the NASA computers. Finally, results are retrieved by TELNET to be processed into graphical displays by PATRAN at the CAE facility. This approach has worked well during 1990, when all of the analyses have been of buckling loads. During 1991, the emphasis will be on thermal stresses.

Progress During 1990

Because a complete buckling analysis of the proposed ALS tank structure would have required far more memory than is available on the CONVEX, a piece-meal approach was taken in which the stringers were evaluated independently, their properties were determined, and then larger elements of the structure were analyzed using beam approximations for the stringers and frames.

In the composite figure, the central figure (1) shows the detailed computer model of the stringer. Starting at the upper left, the figure shows highlights in the comparative analysis of the stringers. First (2), the accuracy of TESTBED was evaluated. For a simply supported panel of 1/4 inch thick aluminum which buckled at 4.787 kips/in according to theory, a panel composed of a 4x6 array of subelements buckled at 4.755 kips/in. Next (3), local buckling loads of both the formed and machined stringers were calculated. Here, the superplastically formed stringer was found to buckle locally above 6 kips/in. Then (4), both stringer designs were analyzed attached to skin panels to determine the proper spacing between rings. In (5), the equivalent stringer properties were evaluated.

The elastic properties of the formed stringers are affected by the corrugations. NASA engineers had determined the effective stringer areas experimentally, but this is the first time that any of the properties have been calculated. One cell of skin, stringer, and ring is shown in (6); this confirmed that the design of the intersection was feasible and that local buckling stresses of the stringers were sufficiently high. Finally, in Figure (7), the stringers were replaced by offset beams using properties that had been obtained under Figure (5), and a section of tank was analyzed for buckling which included one quarter of the circumference of the 30 foot diameter and one quarter of the 30 foot height. When a 28 psi internal pressure was applied, a panel loading of 9.997 kips/in was obtained at buckling. When the torsional stiffness of the stringers was eliminated, this figure dropped to 8.211 kips/in, and

when the internal pressure was eliminated, it dropped further to 5.852 kips/in. In each case, buckling occurred first in the skin panels, rather than in overall buckling of the stringer and frame structure. Interestingly, when the panel is flattened, the load at buckling falls to a negligible value, showing that much of the strength of the tank is due to its curvature.

The significance of this study is that it proved that the proposed superplastically formed stringers and intersecting frames, spot welded to one quarter inch thick sheet, could replace a previously proposed design in which the skin and stringers were to be machined out of solid sheet, one and a quarter inches thick, and were then to be supported by separate frames. The design was intended to carry an ultimate loading of 5.305 kips/in. Thus, even without internal pressure, and ignoring the effects of torsional stiffness of the stringers, there is still a calculated 10% margin of safety. Of course, internal pressure relieves the compressive load on the structure, so that, at 59 psi internal pressure, there would be no compressive load on the structure under ultimate conditions. The traditional method of analysis, which assumes flat panels, would have called for heavy internal ring frames, similar to those on wide-body aircraft. A word of caution, the boundary conditions on a one-sixteenth section of tank cannot fully represent conditions on the full tank, so that some buckling modes could have been eliminated. Also, the analysis makes no allowance for initial imperfections.

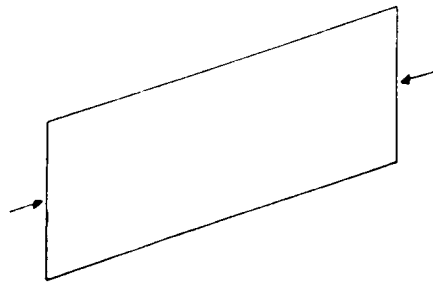
The separate Figure (8) shows a typical buckling analysis of the tank section which involves overall buckling of the stringers and frames. It was determined that this was not the fundamental mode of buckling in the design finally studied, but that the skin panels were the first to buckle. In the figure, it is difficult to distinguish the stringers and frames from the subelement boundaries because offset beams are not reproduced in the graphics.

Plans for 1991

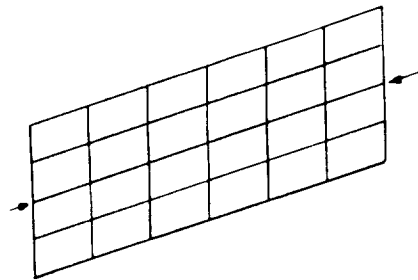
For 1991 we plan to investigate the effects of thermal gradients on the behavior of shell structures in such aerospace vehicles as the high speed civil transport or the National Aerospace Plane (NASP). For example, fuselage sections for high speed civil transports may be subjected to temperature distributions along the fuselage length, and cryogenic fuel tanks for high-velocity aerospace vehicles can have extreme through-the-thickness temperature

gradients. A fundamental study is needed to determine the response of such shell structures subjected to thermal gradients.

In this project, several shell configurations will be considered, including a single-walled shell with external insulation and one with internal insulation to study how the placement of insulation affects the shell. In addition, double walled shells with sandwich material which provides insulation or merely shear strength will be considered. Another configuration will be a shell with heavy internal frames. Each of these configuration studies will include the effects of materials and non-circular cross-sections.



Theory ($N = 4.787$ k/in)

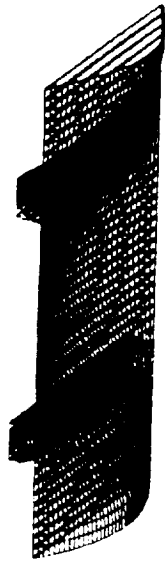


Testbed ($N = 4.755$ k/in)

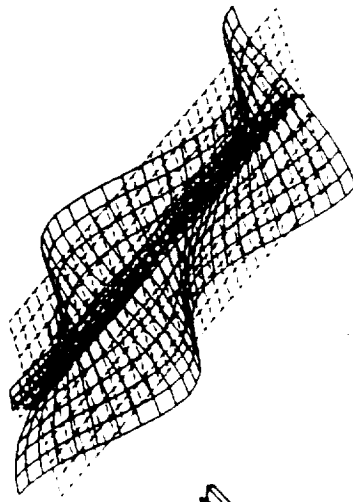
(2) CHECK ON TESTBED ACCURACY FOR SIMPLE PANELS



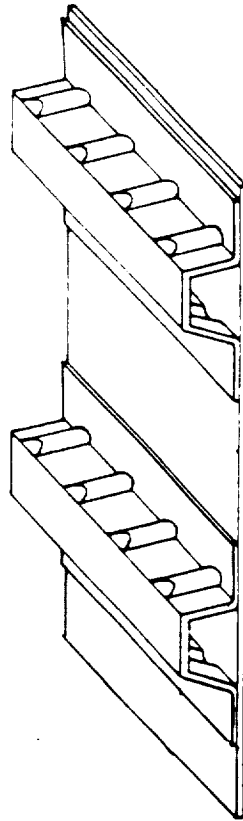
Integral Stringer Machined
out of 1.5" Plate



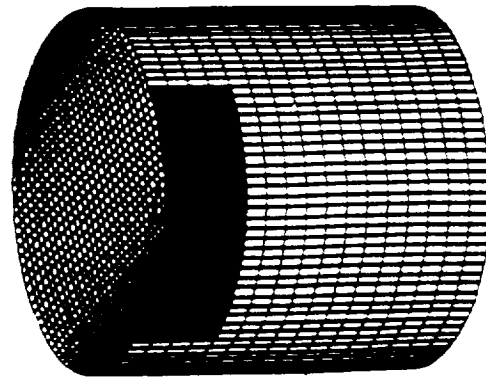
Superplastically Formed
Stringer on 0.25" Plate
(3) COMPARISON OF LOCAL BUCKLING FOR TWO STRINGER DESIGNS



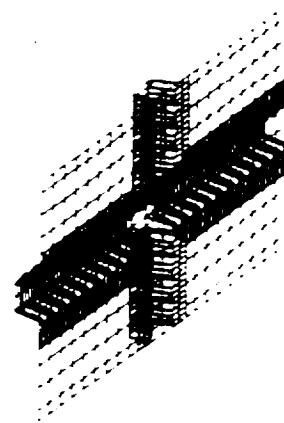
(4) Skin Stringer Analysis



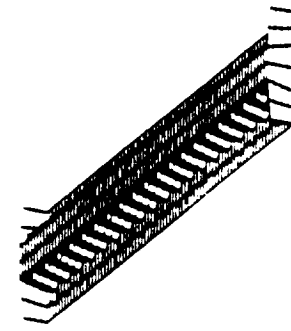
(1) Superplastically Formed Stringer



(7) One Sixteenth of Tank
 $N = 9.497$ k/in



(6) Panel with Intersecting Stringer and Frame



(5) Properties of Stringer

FIGURES 1 TO 7: EVALUATION OF SUPERPLASTICALLY FORMED STRINGERS

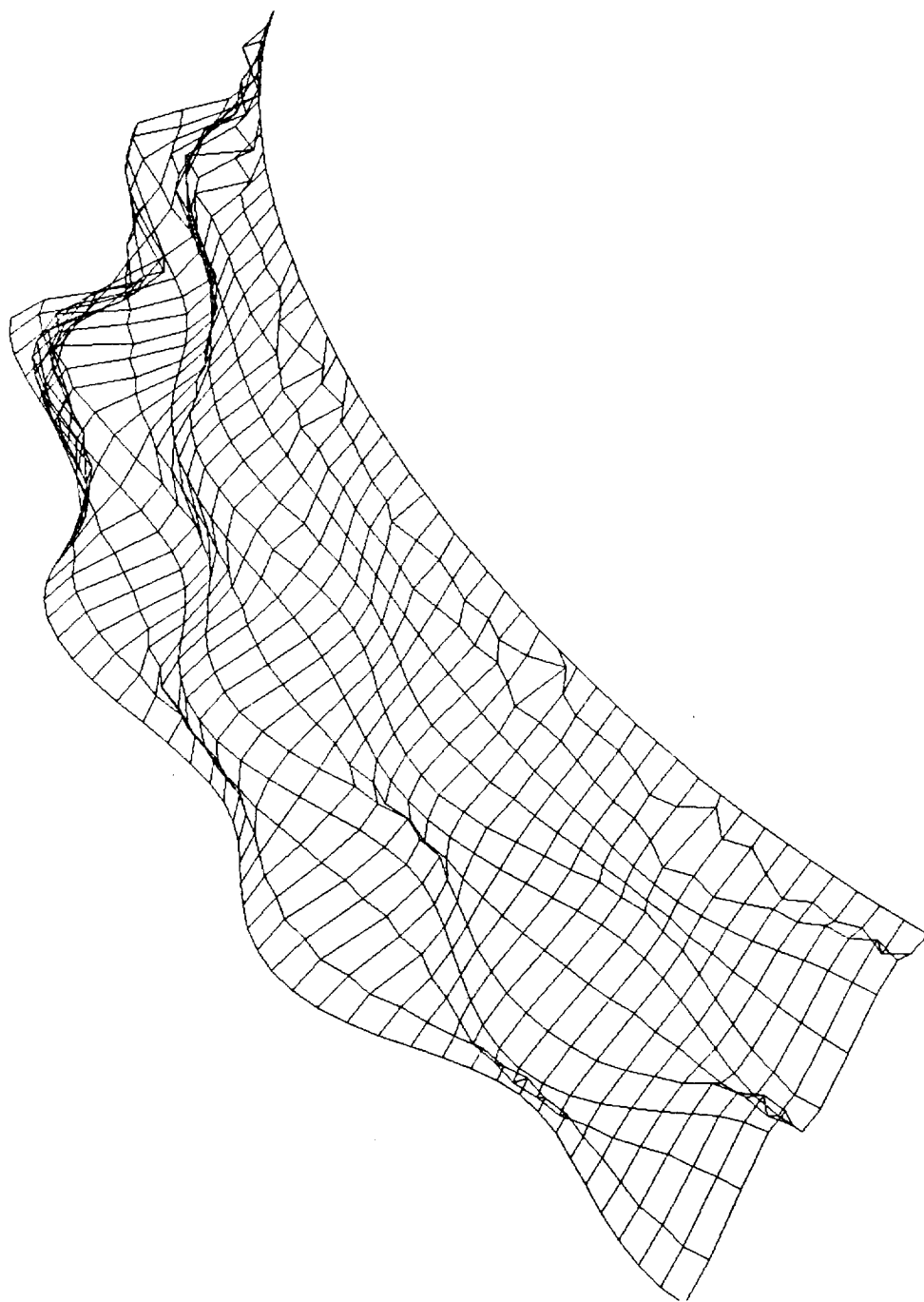


FIGURE 8: BUCKLED SECTION OF TANK

Program 12 Experimental Study of the Viscoplastic Response of High Temperature Structures

Marshall F. Coyle and E.A. Thornton

Objectives

The basic objective of this research program is to investigate experimentally the viscoplastic response of thermal structures for high speed flight. An additional objective of the experimental program is to provide high quality data for validation of finite element analysis using unified viscoplastic constitutive models.

Approach

Simplified structures of representative high temperature superalloys and/or advanced aluminum alloys will be designed and tested. These alloys will display typical biaxial stress states with appropriate temperatures and stress gradients. The simplified structures will provide well-defined thermal and structural boundary conditions.

Research Progress

The first simplified structure tested was a 10 x 15 inch rectangular 304 stainless steel alloy panel with 1/8 inch thickness. The panel was unrestrained. The panel was heated transiently along the centerline and cooled along its edges (Figure 1). Initial tests used resistance heating and tap water for cooling. A nichrome wire bonded to the panel's centerline served as the heating element. The adhesive (RTV) used to bond the element to the panel limited the maximum obtainable temperature. Two edges were cooled by running tap water through PVC pipes bonded to opposite edges of the panel. Each edge of the panel was inserted and bonded in a slot that was machined into a PVC pipe. The tap water's temperature varied slightly throughout the tests. The panel was instrumented with thermocouples to measure in-plane and through the thickness temperatures.

The first experiments were for the purpose of observing qualitatively the physical behavior of the panel. A typical test had an in-plane temperature difference between the

edges of the panel and centerline of 250°F. The thickness temperature difference was less than 3°F. The panel during testing displayed a large out-of-plane bow. The panel displaced approximately 3/8 inch (three times the plate thickness) at the center. Several tests were performed with slight variations. One of the variations was moving the heating element from one side of the panel to the other side. It was found that panel bowing was independent of the side of the panel to which the heating element was attached. Results of preliminary experiments demonstrated in-plane temperature gradients caused transverse bending. This is due to induced compressive membrane stresses acting on an imperfectly flat panel. The bending was not caused by a temperature gradient through the panel thickness.

During the initial test phase, a set of tests were run to determine the effects of insulation. Tests with and without insulation were conducted with the panel oriented horizontally and vertically. The insulated panel had essentially a linear steady-state temperature profile. Without insulation, the panel temperature profile was nonlinear in horizontal orientation and became more nonlinear in the vertical orientation. Figure 2 shows a plot of test results for a panel with and without insulation. This figure shows that the temperature profile of the insulated panel is nearly linear.

Preparations are nearing completion for a second series of tests using Hastelloy-X. This material was selected because material property data at elevated temperatures is available for the Bodner-Partom viscoplastic constitutive model. Ten 10 x 15 inch Hastelloy-X panels with 1/8 inch thickness have been acquired. An extensive series of measurements of initial panel deformations has been performed at NASA Langley. Figure 3 is a typical plot of the panel measurements. It can be seen from Figure 3 that the panel has significant initial deformation with a maximum of plus 0.013 inch and a minimum of negative 0.005 inch. Clearly, the panel is not perfectly flat.

Two panels have been instrumented. One contains thermocouples and the other both thermocouples and strain gages. The transverse displacements of both panels will be measured with LVDTs. Panel No. 1 has 14 strain gages and 20 thermocouples. Figures 4 and 5 show the location of the strain gages and thermocouples. The strain gages have an associated thermocouple for determining the apparent strain. Apparent strain curves were obtained for each of the panel strain gages. The panel was placed in a oven with the

instrumentation attached to a data acquisition system. Initial readings (strain and temperature) were taken at 70°F. The temperature of the oven was raised to 100°F and maintained at this temperature for 30 min. A set of readings were then taken. This allowed for the panel to reach an equilibrium state. This procedure was continued in 50°F increments up to 500°F. The panel was run through several of these thermal cycles.

Panel No. 2 will have 18 thermocouples to measure the in-plane and through the thickness temperature distribution. The thermocouples will have the same locations as panel No. 1 except for thermocouple locations 11 and 14 which were not used.

A test fixture has been designed and fabricated. The fixture supports the panel at four adjustable points. Initially, the supports will be located 3-1/2 inches on either side of the transverse center line and 1-3/4 inches inside the transverse edge. The fixture incorporates a Research Inc. "High-Intensity Infrared Elliptical-Reflector" (Model 5193-16) 16 inch long line heater. The fixture also provides a means for attaching 25 LVDTs to make transverse displacement measurements (see Figure 6). The LVDTs will initially be located along the panel centerline and two adjacent edges. A chilled water system will be used to cool the edges of the panel. This will allow for the coolant temperature to be controlled between 0° and 70°F.

The line heater generates a concentrated radiant heat flux along a thin line. The heat flux drops off rapidly away from the focal line. The heat flux also varies along the focal line. A series of calibration tests were performed to establish the flux field. The line heater was attached to a X-Y indexing mechanism. A calorimeter with an 1/8 inch aperture was suspended above the heater. This allowed the lamp to be indexed under the calorimeter. Figure 7 is a plot of the measured heat flux (mV) at 40% power.

Plans for the Future Research

In the next six months, tests will begin on the two instrumented panels. The first set of experiments will use panel No. 2 which is instrumented with 18 thermocouples. The panel will also be instrumented with 16 LVDTs to measure displacements. A series of tests will be conducted at different heat flux levels. The flux level will start low and will be increased until plastic deformation takes place. This set of tests will be used to validate

experimental techniques and will also give quantitative information on panel behavior. The tests will establish the variation of out-of-plane displacement with temperature.

A second set of experiments will use panel No. 1 which is instrumented with 14 strain gages and 20 thermocouples. This panel will also be instrumented with 16 LVDTs used to measure displacements. This set of experiments will provide strain data in addition to similar information generated in the first set of experiments. A series of tests will investigate the behavior of this panel with increasing temperature levels including inelastic behavior.

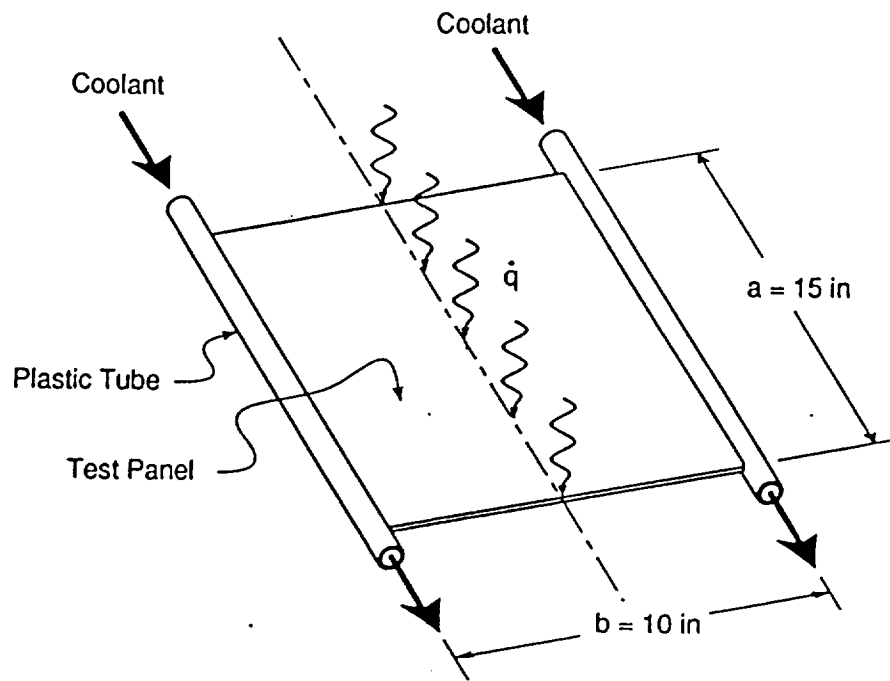


Figure 1: EXPERIMENTAL MODEL

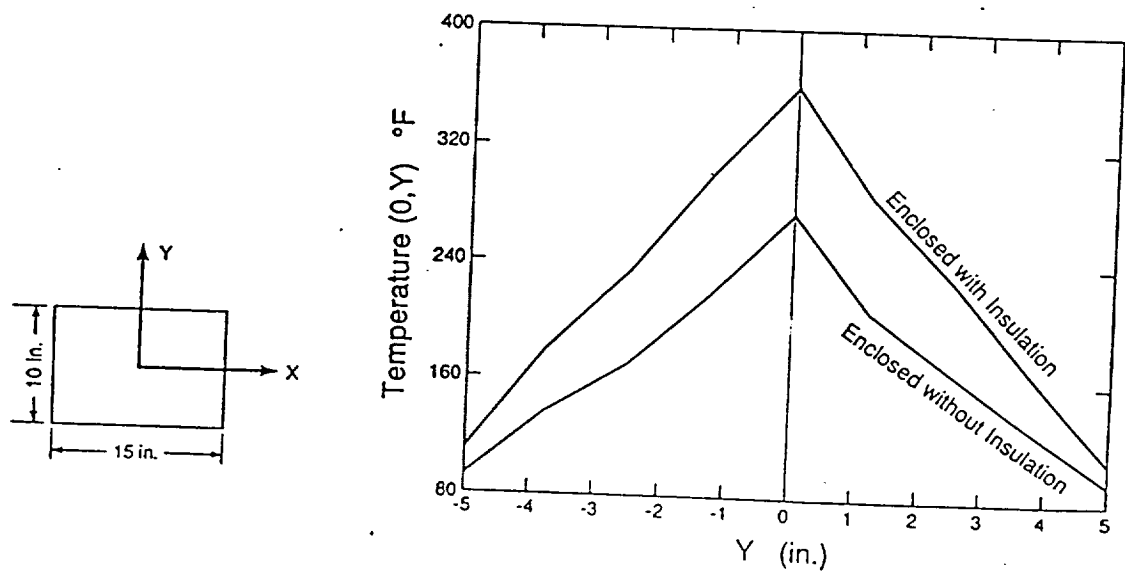


Figure 2: EFFECTS OF INSULATION

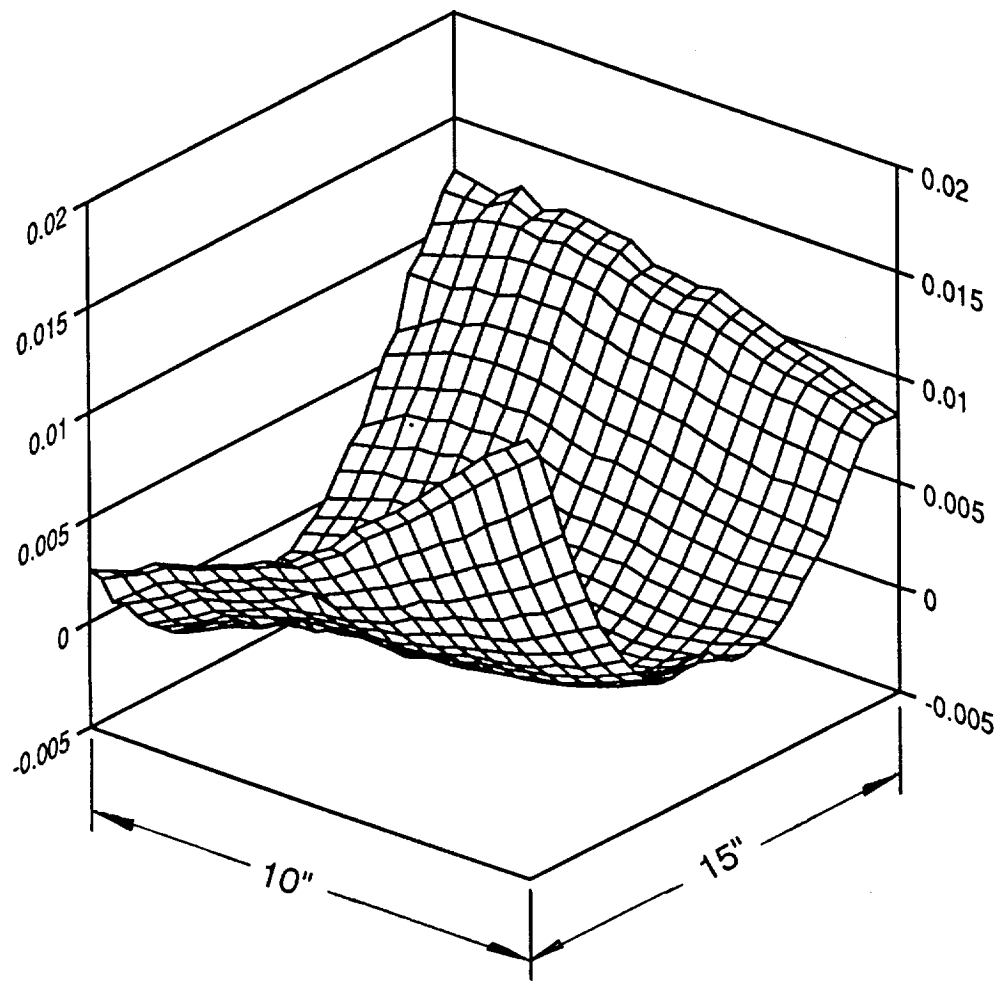


Figure: 3 PANEL MEASUREMENT RESULTS

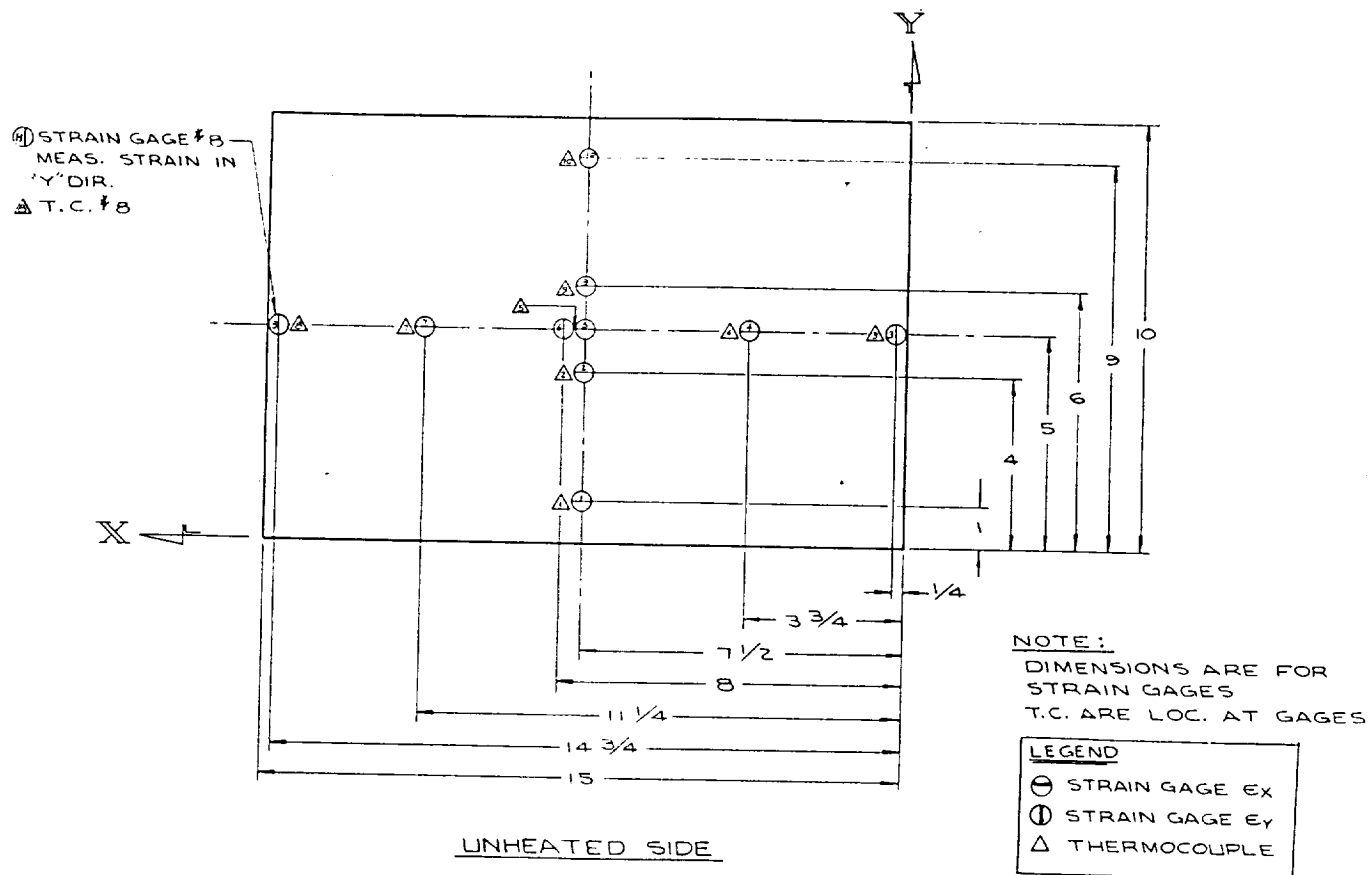


Figure 4: STRAIN GAGE & THERMOCOUPLE LOCATIONS ON UNHEATED SIDE OF PANEL

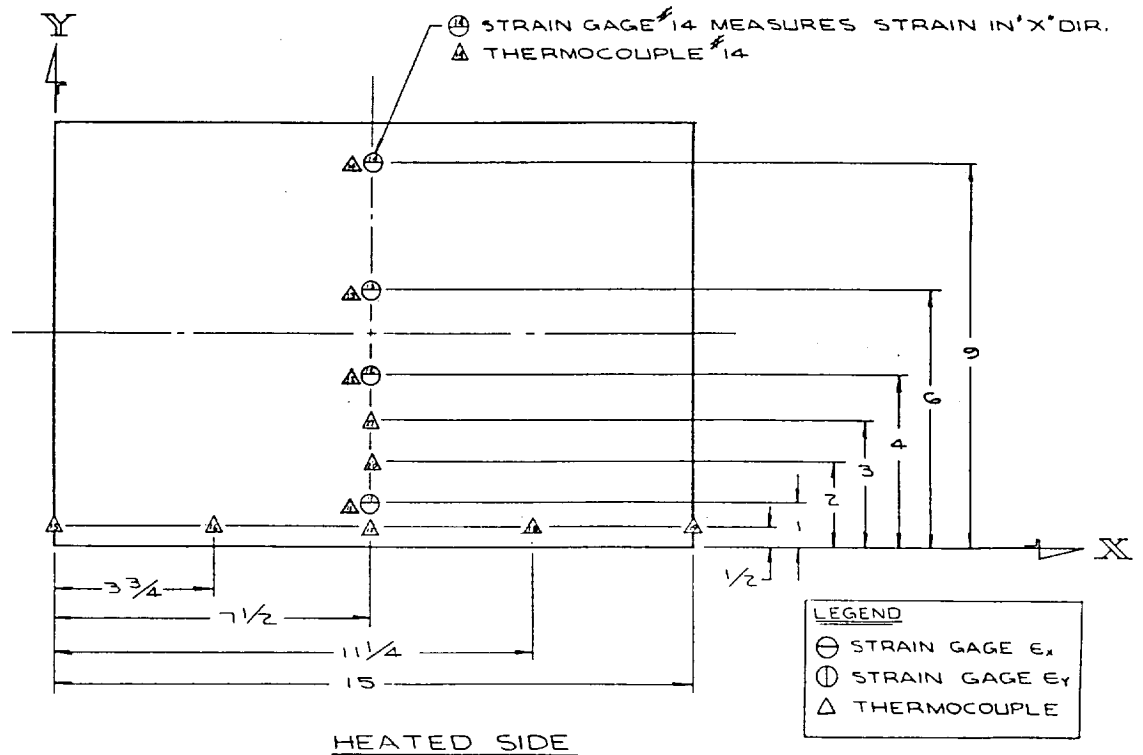


Figure 5: STRAIN GAGE & THERMOCOUPLE LOCATIONS ON HEATED SIDE OF PANEL

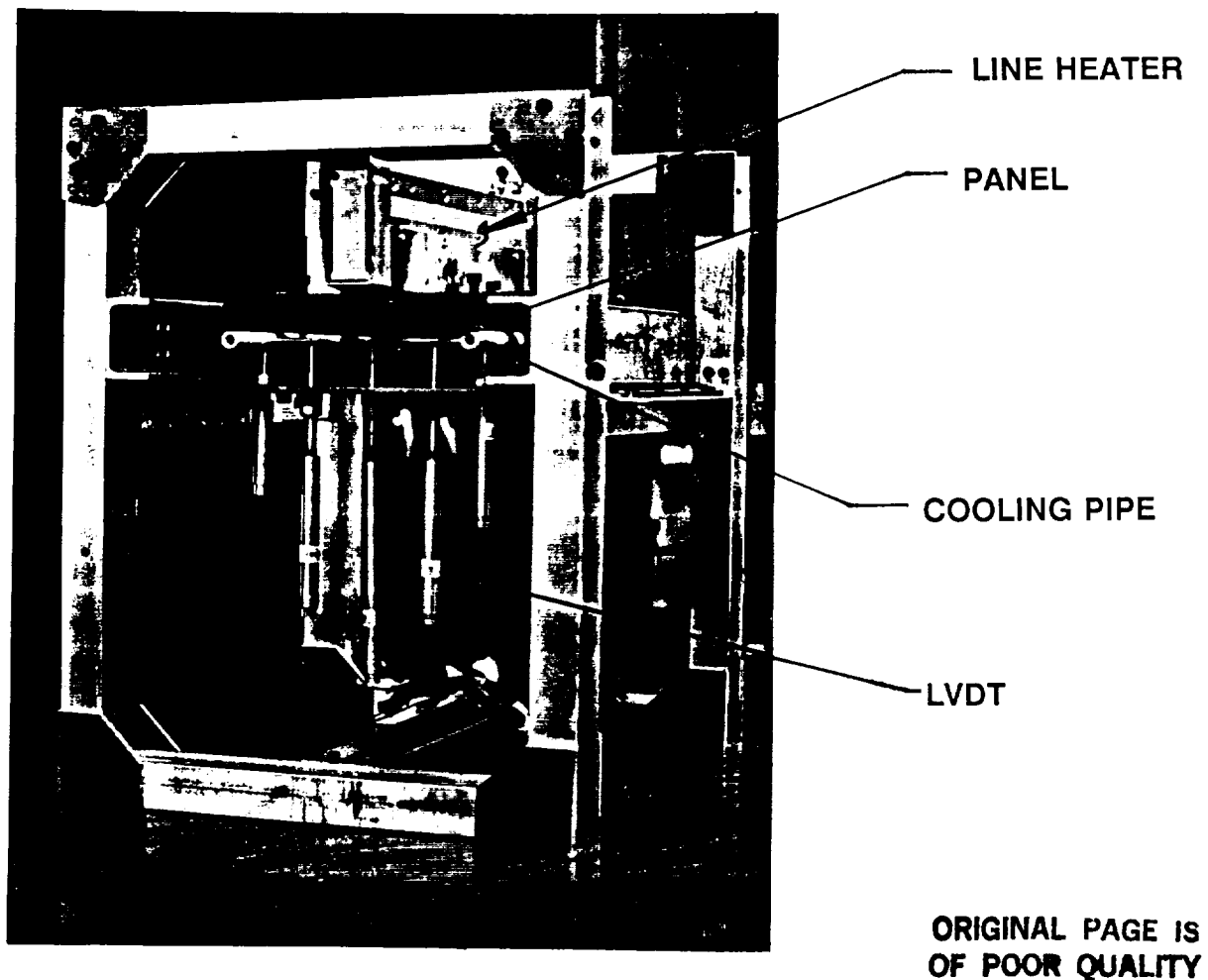


Figure 6: TEST FIXTURE

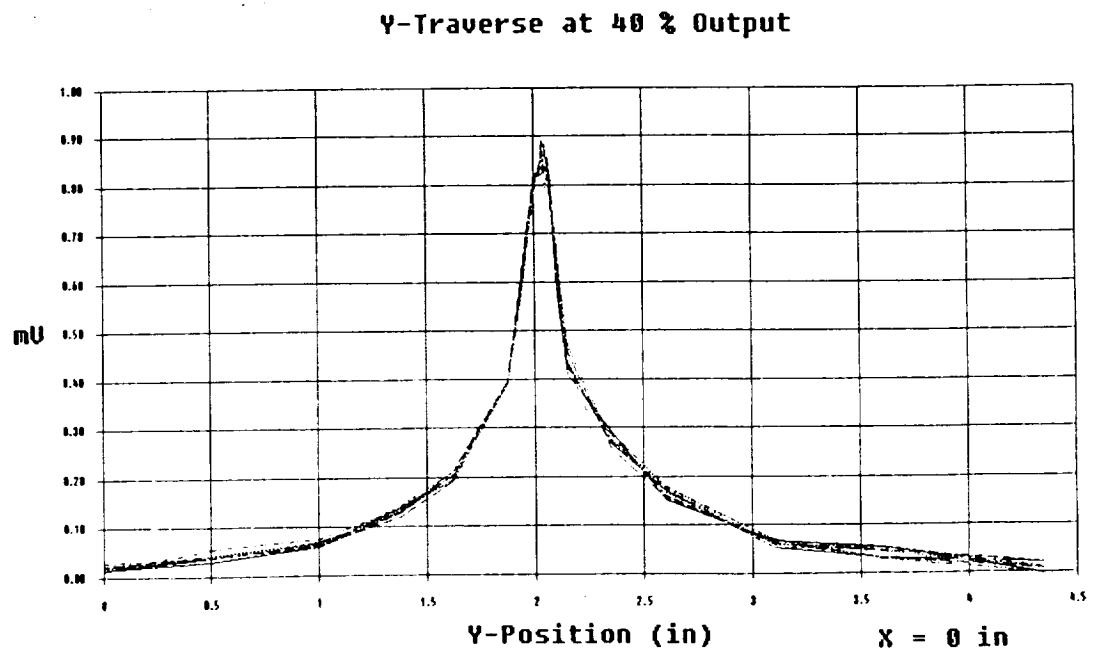


Figure 7: HEAT FLUX MEASUREMENTS

APPENDIX I: GRANT PUBLICATIONS

1. R.P. Gangloff, D.C. Slavik, R.S. Piascik and R.H. Van Stone, "Direct Current Electrical Potential Measurement of the Growth of Small Fatigue Cracks", in Small Crack Test Methods, ASTM STP, J.M. Larsen and J.E. Allison, eds., ASTM, Philadelphia, PA, in review (1991).
2. R.S. Piascik and R.P. Gangloff, "Environmental Fatigue of an Al-Li-Cu Alloy: Part I - Intrinsic Crack Propagation Kinetics in Hydrogenous Environments", Metall. Trans. A, in review (1991).
3. Yang Leng, William C. Porr, Jr. and Richard P. Gangloff, "Time Dependent Crack Growth in P/M Al-Fe-V-Si at Elevated Temperatures", Scripta Metallurgica et Materialia, in press, April (1991).
4. W.C. Porr, Jr., Y. Leng, and R.P. Gangloff, "Elevated Temperature Fracture Toughness of P/M Al-Fe-V-Si", in Low Density, High Temperature P/M Alloys, W.E. Frazier, M.J. Koczak, and E.W. Lee, eds., TMS-AIME, Warrendale, PA, in press (1990).
5. Y. Leng, W.C. Porr, Jr. and R.P. Gangloff, "Tensile Deformation of 2618 and Al-Fe-Si-V Aluminum Alloys at Elevated Temperatures", Scripta Metallurgica et Materialia, Vol. 24, pp. 2163-2168 (1990).
6. R.G. Buchheit, F.D. Wall, G.E. Stoner and J.P. Moran, "Stress Corrosion Cracking of Al-Li-Cu-Zr Alloy 2090 in Aqueous Cl^- and Mixed $\text{CO}_3^{2-}/\text{Cl}^-$ Environments", Paper No. 99, CORROSION/91, Cincinnati, OH, March, 1991.
7. R.G. Buchheit, J.P. Moran, F.D. Wall, G.E. Stoner, "Intersubgranular SCC of Al-Li-Cu Alloy 2090 in Mixed Chloride-Chromate Environments", Conf. Proc. Electrochemical Society, October, Seattle, WA, 1990.
8. R.J. Kilmer and G.E. Stoner, "Effect of Zn Additions on Precipitation During Aging of Alloy 8090", Scripta Metallurgica et Materialia, Vol. 25, pp. 243-248, 1991.

9. F.C. Rivet, "Deformation and Fracture Behavior of Aluminum-Lithium Alloys: The Effect of Dissolved Hydrogen", Master of Science Thesis, Virginia Polytechnic Institute, Blacksburg, VA, 1990.
10. D.B. Gundel and F.E. Wawner, "Interfacial Reaction Kinetics of Coated SiC Fibers with Various Titanium Alloys," Scripta Metallurgica et Materialia, Vol. 25, pp. 437-441, 1991.
11. D.B. Gundel, "Investigation of the Interaction Between Silicon Carbide Fibers and Titanium Alloys," Master of Science Thesis, University of Virginia, Charlottesville, VA, 1991.

APPENDIX II: GRANT PRESENTATIONS

1. R.P. Gangloff, "Fatigue and Fracture Mechanics of Advanced Metallic Alloys", University of Texas at El Paso Lecture Series on Materials Science and Engineering for Manufacturing, November, 1990.
2. R.P. Gangloff, D.C. Slavik, R.S. Piascik and R.H. Van Stone, "Direct Current Electrical Potential Measurement of the Growth of Small Fatigue Cracks", ASTM Symposium on Small Crack Test Methods, San Antonio, TX, November, 1990.
3. W.C. Porr, Yang Leng and R.P. Gangloff, "Elevated Temperature Fracture Toughness of P/M Al-Fe-Si-V", TMS-AIME Symposium on Low Density, High Temperature P/M Alloys, Detroit, MI, September, 1990.
4. W.C. Porr, "Elevated Temperature Fracture Toughness of P/M Al-Fe-Si-V", Poster, First Thermal Structures Conference, University of Virginia, Charlottesville, VA, November, 1990.
5. Yang Leng, "Elevated Temperature Subcritical Crack Growth in Aluminum Alloys", Poster, First Thermal Structures Conference, University of Virginia, Charlottesville, VA, November, 1990.
6. R.G. Buchheit, J.P. Moran, F.D. Wall, G.E. Stoner, "Intersubgranular SCC of Al-Li-Cu Alloy 2090 in Mixed Chloride-Chromate Environments", Fall Meeting of the Electrochemical Society, October 14-19, 1990, Seattle WA.
7. D.B. Gundel and F.E. Wawner, "Investigation of the Reaction Kinetics Between SiC Fibers and Selectively Alloyed Titanium Matrices," in Proceedings of 14th Annual Conf. on Composite Materials and Advanced Structures, Cocoa Beach, FL., January 17-19, 1990.
8. F. Wawner and D. Gundel, "Investigation of Reaction Kinetics and Interfacial Phase Formation in $Ti_3Al + Nb$ Composites," published in Proceedings of Titanium Aluminide Composite Workshop, Orlando, FL, May 16-18, 1990.

9. D.B. Gundel and F.E. Wawner, "Investigation of the Reaction Kinetics Between SiC Fibers and Selectively Alloyed Titanium Matrices," presented at Aeromat '90, Long Beach, CA, May 21-24, 1990.
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11. C.T. Herakovich and J.S. Hidde, "Response of Metal Matrix Composites with Imperfect Bonding", Arizona State University Winter Workshop on the Structure and Properties of Interfaces, Jan. 3-6, 1991.

APPENDIX III: ABSTRACTS OF GRANT PUBLICATIONS

DIRECT CURRENT ELECTRICAL POTENTIAL MEASUREMENT OF THE GROWTH OF SMALL FATIGUE CRACKS

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Reference: R.P. Gangloff, D.C. Slavik, R.S. Piascik and R.H. Van Stone, "Direct Current Electrical Potential Measurement of the Growth of Small Fatigue Cracks", in Small Crack Test Methods, ASTM STP, J.M. Larsen and J.E. Allison, eds., ASTM, Philadelphia, PA, in review (1990).

ENVIRONMENTAL FATIGUE OF AN Al-Li-Cu ALLOY: PART I - INTRINSIC CRACK PROPAGATION KINETICS IN HYDROGENOUS ENVIRONMENTS

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ABSTRACT

Deleterious environmental effects on steady-state, intrinsic fatigue crack propagation (FCP) rates (da/dN) in peak aged Al-Li-Cu alloy 2090 are established by electrical potential monitoring of short cracks with programmed constant ΔK and K_{max} loading. da/dN are equally unaffected by vacuum, purified helium and oxygen; but are accelerated in order of decreasing effectiveness by aqueous 1% NaCl with anodic polarization, pure water vapor, moist air and NaCl with cathodic polarization. While da/dN depends on $\Delta K^{4.0}$ for the inert gases, water vapor and chloride induce multiple power-laws and a transition growth rate "plateau". Environmental effects are strongest at low ΔK . Crack tip damage is ascribed to hydrogen embrittlement because of: accelerated da/dN due to part-per-million levels of H_2O without condensation, impeded molecular flow model predictions of the measured water vapor pressure dependence of da/dN as affected by mean crack opening, the lack of an effect of film-forming O_2 , the likelihood for crack tip hydrogen production in NaCl, and the environmental and ΔK -process zone volume dependencies of the microscopic cracking modes. For NaCl, growth rates decrease with decreasing loading frequency, with the addition of passivating Li_2CO_3 and upon cathodic polarization. These variables increase crack surface film stability to reduce hydrogen entry efficiency. Small crack effects are not observed for 2090; such cracks do not grow at abnormally high rates in single grains or in NaCl, and are not arrested at grain boundaries. The hydrogen environmental FCP resistance of 2090 is similar to other 2000 series alloys and is better than 7075.

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TIME DEPENDENT CRACK GROWTH IN P/M Al-Fe-V-Si AT ELEVATED TEMPERATURES

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Introduction

Time dependent subcritical crack growth can be an important fracture mode which affects the damage tolerance of structural materials in elevated temperature service [1,2]. While referred to as "creep crack growth" (CCG), the causal micromechanism may include creep deformation, microstructural instability and environmental embrittlement due to oxygen or hydrogen.

It is critical, from both the life prediction and mechanistic perspectives, to determine the correct continuum fracture mechanics parameter which describes the governing crack tip stress, strain and strain rate fields [1,2]. For creep ductile materials (e.g. alloy steels), time dependent crack growth is reasonably characterized by the C -integral under steady-state creep conditions and by C_1 under small scale creep (SSC) and transient creep (TC) conditions [1,2]. For creep brittle materials (e.g. Ni and Ti alloys), time dependent crack growth is governed by time independent parameters such as the small scale yielding stress intensity (K) or the elastic-plastic J -integral [3,4].

Elevated temperature crack growth occurs in aluminum alloys, however, such behavior has not been broadly defined, particularly for advanced materials with thermally stable ultra-fine grain size microstructures [5-7]. For the latter, recent results indicate loading rate dependent fracture toughness, suggesting that time dependent subcritical crack growth is important [8]. This report presents observations of time dependent crack growth in rapidly solidified alloy 8009 (previously called FVS0812) designed for elevated temperature applications [9]. The applicability of several fracture mechanics parameters (viz. C , C_1 , K and J) to correlate crack growth kinetics is investigated.

Experimental Procedure

Powder processed, hot extruded 8009 (Al-8.5Fe-1.3V-1.7Si (wt%)) plate was supplied by Allied-Signal, Inc. Rapid solidification processing produces a microstructure with ultra fine $Al_{12}(Fe,V)_3Si$ dispersoids (less than 0.1 μm in size) distributed in the aluminum matrix of grain size less than 1 μm [8,9].

Sidegrooved compact tension (CT) specimens in the L-T orientation (width (W) = 38.1 mm, gross thickness = 7.62 mm, net thickness = 6.35 mm) were used for crack growth experiments. Experiments were conducted with a servo-electric mechanical testing system. Specimens were instrumented to measure load (P), front-face crack mouth opening displacement (CMOD), crack length (a) from electrical potential and temperature, as described in Ref. 8. Specimens, fatigue precracked to an a/W of 0.5, were tested at 175, 250 and 316°C under constant load. Load levels were chosen to be approximately 40% of the load at which the "fast loading rate" K_{IC} is defined [8]. Crack growth rate was determined from crack length versus time measurements. The minimum crack extension between successive data points was about 0.02 mm and the secant method was used to obtain the crack growth rate (da/dt). A JEOL JXA-840A scanning electron microscope (SEM) and a Phillips EM 400T transmission electron microscope (TEM) were used for fractography and microstructural examinations. To determine power-law creep constants, tensile creep deformation tests were conducted at 175, 250 and 316°C in a dead weight creep machine. The round bar specimens were 5.7 mm in diameter. The tensile strain was measured with a high temperature Capacitec extensometer over a gage length of 25.4 mm.

Results and Discussion

Experimental observations indicate that 8009 exhibits substantial time-dependent subcritical crack growth at each of the test temperatures, with the rates varying between 0.003 and 0.2 mm/hour. Crack growth occurs at stress

ELEVATED TEMPERATURE FRACTURE TOUGHNESS OF P/M Al-Fe-V-Si

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Abstract

The effects of temperature and loading rate on the crack initiation and growth fracture toughnesses of powder metallurgy Al-8.5Fe-1.7Si-1.3V (wt%) were investigated by an elastic-plastic J-integral method with electric potential difference crack monitoring. At 25°C the ultra-fine grained P/M alloy exhibits high crack initiation toughness ($K_{IC} = 36.6$ MPa/m) and crack growth resistance ($T_R = [E/\sigma_o^2][dJ/da] = 20.6$) in the LT orientation compared to the behavior of ingot metallurgy alloy 2618 ($K_{IC} = 21$ MPa/m and $T_R = 0.5$). While the toughness of the I/M alloy is constant (K_{IC}) or increases mildly (T_R) with temperature to the aging level of 199°C, both measures of toughness for the P/M alloy decrease between 25°C and 316°C, with minima in K_{IC} and T_R at 200°C. K_{IC} decreases and T_R increases mildly with increasing temperature for FVS0812 tested in the TL orientation. K_{IC} and T_R decrease with decreasing applied load line displacement rate for LT FVS0812 at 175°C, with crack growth resistance exhibiting a minimum. The mode of fracture is microvoid coalescence in all cases, however, the origins and evolution of damage are unclear. The TL toughness of FVS0812 is generally low because of prior particle boundary oxides oriented parallel to the fracture path. The behavior of LT FVS0812 is explained by a complex interaction of prior particle boundary delamination, perpendicular to the main crack front to promote plane stress "thin sheet toughening", and several possible intrinsic fracture mechanisms. Speculatively, intrinsic toughness decreases with increasing temperature and loading time because of dynamic strain aging from solid solution Fe or environmental hydrogen embrittlement.

W.C. Porr, Jr., Y. Leng, and R.P. Gangloff, "Elevated Temperature Fracture Toughness of P/M Al-Fe-V-Si", in *Low Density, High Temperature P/M Alloys*, W.E. Frazier, M.J. Koczak, and E.W. Lee, eds., TMS-AIME, Warrendale, PA, in press (1990).

TENSILE DEFORMATION OF 2618 AND Al-Fe-Si-V ALUMINUM ALLOYS AT ELEVATED TEMPERATURES

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Introduction

Quantitative descriptions of the temperature dependent elastic-plastic deformation of advanced aluminum alloys are critical, but unexplored. Such alloys are processed by deformation consolidation of rapidly solidified or mechanically alloyed powders to develop sub-micron grain sizes and a dispersion of small (50 nm) particles (1,2). These novel microstructures are substantially different from conventional precipitation hardened aluminum alloys. Important issues include quantitative stress-strain relations, heterogeneous yielding, flow localization, strain hardening or softening, strain aging and dislocation morphologies (1-5). In this study the effects of elevated temperature on the uniaxial tensile behavior of ingot metallurgy (I/M) aluminum alloy 2618 and rapidly solidified powder metallurgy (P/M) alloy FVS0812 are experimentally characterized with two constitutive formulations, the Ramberg-Osgood equation and the Bodner-Partom model (6,7). Multi-axial behavior and time dependent plasticity are not considered here.

Stress-Strain Relationships

The *Ramberg-Osgood* (RO) equation empirically describes material constitutive behavior by a power-law relation between true stress (σ) and true total strain (ϵ_t), and which includes elastic and plastic terms (6):

$$\epsilon_t/\epsilon_0 = \sigma/\sigma_0 + \alpha (\sigma/\sigma_0)^n \quad (1)$$

σ_0 is a reference stress, not necessarily equal to engineering yield strength (σ_{y0}), ϵ_0 is a reference elastic strain given by σ_0/E , E is Young's modulus, α is a constant and n is the strain hardening coefficient.

Three parameters must be experimentally determined in the RO equation. n is obtained by least squares analysis of measured load-specimen gauge displacement data logarithmically plotted as true plastic strain ($\epsilon_p = \epsilon_t - \sigma/E$) versus σ from Eq. 1:

$$\ln(\epsilon_p E) = [\ln \alpha - (n-1) \ln \sigma_0] + n \ln \sigma \quad (2)$$

σ_{y0} is used as an initial estimation for σ_0 and α is determined from Eq. 1 by a computer curve fit which iteratively defines the best σ_0 - α pair.

Bodner and Partom (BP) developed a constitutive relation and material constants based on microstructural quantities (8). For uniaxial stress and small strain, the plastic strain rate ($d\epsilon_p/dt$) is expressed as:

$$d\epsilon_p/dt = (2 D_0/\sqrt{3}) (\sigma/|\sigma|) \exp[-0.5 (Z/\sigma)^{2n}] \quad (3)$$

D_0 is the limiting shear strain rate and is taken as a large value (10^4 s^{-1}) for all metals; n' relates to strain rate sensitivity and the intrinsic viscosity of dislocation motion (9); $\sigma/|\sigma|$ gives a negative strain rate for compressive stress; and Z represents the resistance to plastic deformation caused by microstructural barriers impeding dislocations:

$$Z = Z_1 - (Z_1 - Z_0) \exp(-m W_p) \quad (4)$$

W_p is the plastic work to produce a given stress and strain (calculated by the integrated area defined by the σ - ϵ relationship), m is a material constant related to the rate of strain hardening, and Z_0 and Z_1 are the initial and saturation values of Z , respectively. Stress is calculated incrementally from:

$$\sigma = E (\epsilon_{\text{total}} - \epsilon_p) \quad (5)$$

and plastic strain given by:

$$\epsilon_p = \sum (d\epsilon_p/dt) \Delta t \quad (6)$$

Five material parameters; n' , m , D_0 , Z_0 and Z_1 ; must be determined to describe elastic-plastic deformation.

Localized Corrosion Behavior of Alloy 2090—The Role of Microstructural Heterogeneity [☆]

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ABSTRACT

The pitting and intergranular corrosion behavior of alloy AA⁽¹⁾ 2090 (Al-2Li-3Cu, UNS⁽²⁾ A92090) in a 3.5 wt% NaCl solution was investigated. Techniques used included potentiodynamic polarization, galvanic couples, and pH measurements in simulated crevices. Polarization scans were performed on under-aged and peak-aged material to obtain the standard polarization parameters. Corroded specimens were examined with optical microscopy, scanning electron microscopy (SEM), and energy-dispersive x-ray spectroscopy (EDAX) to distinguish various local corrosion morphologies. Ingots were cast to approximate the subgrain boundary, T_1 (Al_2CuLi) phase, and Al-Cu-Fe constituent phases inherent in the alloy. These were then galvanically coupled to solution heat treated (SHT) 2090 to identify their role in local corrosion processes. Simulated crevices were produced by inserting pH micro-electrodes into crevices machined in 2090 blocks to measure pH versus time response in occluded environments.

Based on the experiments listed above, two different types of pitting mechanisms were identified. The first type was directly attributed to the dissolution of the subgrain boundary phase T_1 . A direct correlation between increased subgrain boundary precipitation and increased subgrain boundary pitting was observed. The second type of pitting involved enhanced local galvanic attack of the matrix material surrounding Al-Cu-Fe constituent particles found in this plate. With this type of attack, large pits formed around constituents which occurred randomly throughout the plate.

Associated with constituent particle pitting was a form of localized attack that was designated as continuous subgrain boundary dissolution. This form of corrosion was observed on all specimens used in corrosion experiments. It was only seen, however, in regions adjacent to the largest pits on the specimen, suggesting the influence of an occluded (crevice) environment. Simulated crevice experiments revealed that an acidic crevice solution developed with time. This environment is suspected to contribute to the continuous subgrain boundary dissolution in the vicinity of the constituent particles.

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KEY WORDS: aluminum-lithium alloys, intergranular corrosion, localized corrosion, microstructural heterogeneity, pitting

INTRODUCTION

Aluminum-lithium-X alloys are susceptible to localized corrosion in chloride-containing aqueous environments. Localized corrosion manifests itself as stress corrosion cracking (SCC), crevice corrosion, and pitting. All three forms of environmental attack have been observed in these alloys and microstructural heterogeneity is often implicated as a contributing factor.¹⁻⁶

An emergent class of Al-Li-Cu alloys are being studied as possible substitutes for the more conventional 2XXX and 7XXX series alloys, particularly in applications where weight savings are a premium. It is well known that additions of lithium to aluminum reduce density while increasing modulus.⁷ Although prototype Al-Li-X alloys suffered from unacceptably low ductility and fracture toughness, the commercially produced 2090 (Al-Li-Cu) can achieve acceptable mechanical properties when correctly processed. The mechanical and physical properties of the Al-Li-X system have been well documented;⁷⁻¹⁸ however, a brief review of the physical metallurgy of alloy 2090, with emphasis on those features pertinent to the present study, is appropriate here. The following discussion is schematically summarized in Figure 1.

The primary matrix phases are δ' (Al_3Li), θ' (Al_2Cu), and T_1 (Al_2CuLi). Coherent δ' is observed as discrete particles and as a coating on the broad faces of θ' .^{6,10} The partially coherent T_1 and θ' precipitate heterogeneously along dislocations within the matrix or at Guinier-Preston zones,¹¹⁻¹³ and are primarily responsible for improved ductility, relative to the binary alloy. Both phases enhance cross-slip, helping to combat the localized deformation due to shearing of δ' .¹³⁻¹⁵ T_1 is also the primary phase observed along low-angle (or subgrain) boundaries.^{6,12,13,16} Although the crystallographic nature and exact composition of the high-angle (or grain) boundary phase(s) is somewhat controversial,^{12,16,18} the issue is sufficiently clear from a corrosion standpoint. The primary grain boundary phase resembles (or closely resembles) the non-coherent T_2 (Al_6CuLi_3). The binary equilibrium phase, δ (AlLi), is not observed at typical aging temperatures.^{13,17}

The relative concentrations of these phases is dependent upon aging time. The metastable δ' nucleates immediately upon quenching and reaches a maximum volume fraction well before the peak-aged condition.⁸ Subgrain boundaries are the initial site for T_1 nucleation. Alloys subjected to deformation prior to aging have a higher percentage of T_1 precipitation occurring within the

STRESS CORROSION CRACKING OF AL-LI-CU-ZR ALLOY 2090
IN AQUEOUS Cl^- AND MIXED $\text{CO}_3^{2-}/\text{Cl}^-$ ENVIRONMENTS

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ABSTRACT

A comparison of the short-transverse SCC behavior of 2090 in pH 5.5 Cl^- and alkaline $\text{CO}_3^{2-}/\text{Cl}^-$ solutions using a static load smooth bar SCC technique was made. In the alkaline $\text{CO}_3^{2-}/\text{Cl}^-$ solutions, E_{br} for the α -Al matrix phase was 0.130 V more positive than the E_{br} of the subgrain boundary T_1 phase. In this environment, stress corrosion cracking test specimens subjected to potentials in the window defined by the two breakaway potentials failed along an intersubgranular path in less than an hour. In the Cl^- environment, the E_{br} values for the two phases were nearly equal and this rapid SCC condition could not be satisfied; accordingly SCC failures were not observed.

Rapid SCC failure of 2090 in $\text{CO}_3^{2-}/\text{Cl}^-$ in our static load, constant immersion experiments appear to be related to recently reported "pre-exposure embrittlement" failures induced by immersing stressed specimens removed into ambient laboratory air after immersion in aerated NaCl solution for 7 days. In those experiments, specimens failed in less than 24 hours after removal from solution. Our polarization experiments have shown that the corrosion behavior of T_1 , residing at the crack tip, is relatively unaffected by the alkaline $\text{CO}_3^{2-}/\text{Cl}^-$ environments, but the α -Al phase crack walls, is rapidly passivated. X-ray diffraction of the films which formed in simulated

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EFFECT OF Zn ADDITIONS ON PRECIPITATION DURING AGING OF ALLOY 8090

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Introduction

Al-Li base alloys have received considerable attention as potential lightweight replacements for conventional Al-base alloys in aerospace applications. The low density and high stiffness of alloy 8090 (Al-Li-Cu-Mg) earmarks it as an especially attractive candidate. Alloy 8090 derives its strength from the co-precipitation of Al_3Li (δ'), Al_2CuMg (S') and Al_2CuLi (T_1) during artificial aging. Stretching prior to aging enhances the heterogeneous precipitation of S' and T_1 , greatly improving the fracture toughness properties in Al-Li-Cu-Mg alloys (1). In applications where a pre-aging stretch is not feasible, additions of Zn to the Al-Li-Cu-Mg system have been investigated as a means to improve toughness (2).

The present paper reports the precipitation events of three stretched alloys whose compositions fall within the 8090 composition window and contain varying amounts of Zn additions up to 1.07 wt %. The alloys were examined under two different aging conditions using transmission electron microscopy (TEM) and the results were compared with those of alloy 8090 employed as a baseline. Differential scanning calorimetry (DSC) was employed to provide further insight into the effects that varying the Zn content has on the precipitation events.

Experimental Procedure

The compositions (in wt %) of the wrought alloys used in the present investigation are shown in Table 1. The alloys were solution heat treated for 1 hour at 543C followed by a cold water quench. The alloys were specialty cast by Alcoa, scalped, forged and rolled to 2.5mm sheet. The four alloys were stretched to obtain a T3 condition. The aging hardening characteristics were traced by Vickers pyramid hardness measurements utilizing a 500 gm load with the stress axis normal to the longitudinal direction of the sheet.

Microstructural studies were performed by TEM on the samples in both the as-received (T3) condition and after aging for 100 hours at 160C. This aging time and temperature represents the peak aged condition based on hardness tests. Specimens for TEM were prepared using standard methods and examined with a Phillips 400T electron microscope operating at 120 kV. Thermal analysis of the alloys was undertaken in a Perkin-Elmer DSC using standard procedures immediately after quenching from solution heat treatment utilizing a constant heating rate of 10C/min.

TABLE 1. Chemical compositions of alloys (wt-%)

Alloy code	Li	Cu	Mg	Zn	Zr
A 8090 Baseline	2.44	1.06	0.63	...	0.10
B 8090 + 0.21 Zn	2.27	1.07	0.60	0.21	0.10
C 8090 + 0.58 Zn	1.91	1.07	0.62	0.58	0.10
D 8090 + 1.07 Zn	2.09	1.00	0.59	1.07	0.10

INTERFACIAL REACTION KINETICS OF COATED SiC FIBERS WITH VARIOUS TITANIUM ALLOYS

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Introduction

The high temperature strength and modulus of titanium alloys can be markedly improved by reinforcement with silicon carbide fibers. The resulting composite also has a lower density than the monolithic material, thus making it attractive for aerospace applications. During fabrication and elevated temperature exposure, however, a reaction occurs between the fiber and the matrix which can be detrimental to the mechanical properties of the composite (1-3). The reaction results in the growth of a layer of brittle reaction products (the reaction zone) at the fiber-matrix interface. The reaction zone growth kinetics can be modelled with a parabolic growth law (1,3,4):

$$X = kt^{\frac{1}{2}} + b$$

Here, X is the reaction zone thickness, k is the temperature dependent rate constant, t is the isothermal exposure time, and b is the X -intercept of an X versus $t^{\frac{1}{2}}$ plot. Furthermore, k follows the Arrhenius relation:

$$k = k_0 e^{-\frac{Q}{RT}}$$

Where k_0 is the pre-exponential factor, Q is the apparent activation energy, R is the gas constant, and T is the exposure temperature. Previous researchers have shown that in many titanium alloy/silicon carbide fiber systems the reaction zone consists mainly of titanium carbide and titanium silicides (5-7).

Several studies have demonstrated that alloy additions to titanium can have a strong influence on the rate of the reaction (4,8,9). The purpose of this study is to compare the reaction kinetics of several titanium-based alloys, including Ti-1100 (Ti-6Al-2.8Sn-4Zr-4Mo-45Si-0.07O₂-0.02Fe) and BETA 21S (Ti-15Mo-2.7Nb-3Al-2Si), with SCS-6 fibers in an attempt to determine which may be best suited to high temperature use. SCS-6 is a coated silicon carbide fiber produced by Textron Specialty Materials (Lowell, MA) which uses chemical vapor deposition and is the current fiber of choice for titanium matrices. Ti-1100 is a near alpha alloy developed for elevated temperature use up to 593°C (10), while BETA 21S is a metastable beta alloy that has similar properties to, but improved oxidation resistance over, Ti-15V-3Cr-3Al-3Sn (Ti-15-3) (11). Both Ti-1100 and BETA 21S are produced by Timet (Henderson, NV), and are currently regarded as viable candidates for composite matrices.

Experimental

SCS-6 fiber was incorporated into Ti-1100, BETA 21S, unalloyed (UA) titanium (99.7% metallicly pure), Ti-6Al-4V (Ti-6-4), Ti-15-3, and Ti-14(wt%)Al-21(wt%)Nb (an $\alpha_2 + \beta$ alloy), by diffusion bonding in a vacuum hot-press with a pressure of 103 MPa for 30 minutes. The fabrication temperatures ranged from 850 to 1050°C and were chosen to minimize the as-fabricated reaction zone thickness (typically about 0.5 μ m), but still insure complete consolidation.

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